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PROGRAM, HIGH-TEMPERATURE NITRIDING
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MATERIAL EVALUATION PROGRAM,
HIGH-TEMPERATURE NITRIDING
ENVIRONMENT

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FOREWORD

This report covers a program conducted as a supplemental task within a 12-month NASA gas generator technology contract, NAS9-13003.

Acknowledgment is given to Messrs. R. Binsley, A. Jacobs, J. Hill, W. T. Chandler, and T. MacNamara of Rocketdyne for their technical contribution to this project.

ABSTRACT

The results of a program conducted to evaluate materials for construction of a Space Shuttle hydrazine monopropellant gas generator are presented in this report. The program was designed to select those materials that maintain the properties of strength and ductility after exposure to an 1800 F nitriding environment for 1000 hours.

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INTRODUCTION

The decomposition of hydrazine in a gas generator produces ammonia, which in turn dissociates through surface contacts to form nitrogen. This nitrogen diffuses into the alloy and combines with the alloying elements to form nitrides that act to harden, and adversely affect other material properties, such as strength, elongation, and fatigue characteristics. In the Space Shuttle APU hydrazine gas generator, metal surfaces are subjected to ammonia at temperatures of 1800 F for long periods of time. These high temperatures and long durations not only reduce material strength, but also increase the nitriding process.

As a supplemental task within a NASA gas generator technology contract, a program was initiated to evaluate gas generator material candidates, by subjecting the materials to a 1800 F nitriding environment for 1000 hours.

SUMMARY AND CONCLUSIONS

All materials studied experienced nitriding to some degree after exposure to a simulated 1800 F hydrazine decomposition environment. Nitriding caused a change in the grain structure of the material specimens and in such properties as hardness, ductility, and strength. An increase in both weight and thickness of the specimens was also evident.

Of those materials investigated which are suitable for use in the gas generator, that is, those that are weldable and have a yield strength in excess of 4000 psi, the INCO 600 was least degraded by the 1800 F nitriding environment. A yield strength in excess of 8000 psi and a 50-hour rupture strength in excess of 5000 psi are desirable for a material for use as the structural wall of the gas generator. For this purpose, INCO 617 demonstrated the least loss of ductility and high-temperature elongation.

Results of the high-temperature tensile tests, room-temperature bend tests, and microstructure analysis of the material specimens are summarized in Table 1. None of the high-strength materials would provide a suitably high confidence level for long-duration use in an 1800 F nitriding environment. A redesign of the gas generator was mandatory to reduce the maximum structural wall temperature to 1600 F, and hence decrease the material property degradation due to nitriding. This was achieved by a dual-wall, exhaust gas regenerative design shown in Fig. 1. INCO 600 was selected for the low-stressed thermal bed screens and liner, and INCO 617 was selected for the structural chamber wall and injector. Haynes 188 and L605 were considered a second-best choice for the redesigned gas generator structural wall, which is exposed to reduced gas temperatures of 1400 to 1600 F.

TABLE 1. SUMMARY - MATERIAL NITRIDING STUDY

Specimen	1800 F field Strength (psi)	1800 F Elongation (percent)	Room Temperature Ductility (Bend Angle)	Hardness Percent Increase at Depth >0.010 inch	Comments
		1000 Hours Test Results		100 Hours	
INCO 600	8,100	14.0	180 (crack)	3.0	Flaking of specimen.
INCO 617	21,500	18.0	25-30 (broke)		
Haynes 188	23,100	7.5	0 (broke)	8.0	
L605				18.0	
Screens (INCO 600)			180 (no crack)		3.5-percent increase in weight
Screen Pack (INCO 600)					2-percent increase in weight
		400 Hours Test Results			
Multimet	15,700	52.0	0 (broke)		100-hours specimen snapped when tapped at room temperature
MAR-M-509	26,700	12.0	0 (broke)		Cast alloy
MAR-M-246	45,400	2.0	10-15 (broke)		Cast alloy, non- weldable
TD NI	3,600	38.0	180 (no crack)		Too soft (welding results in loss of strength)

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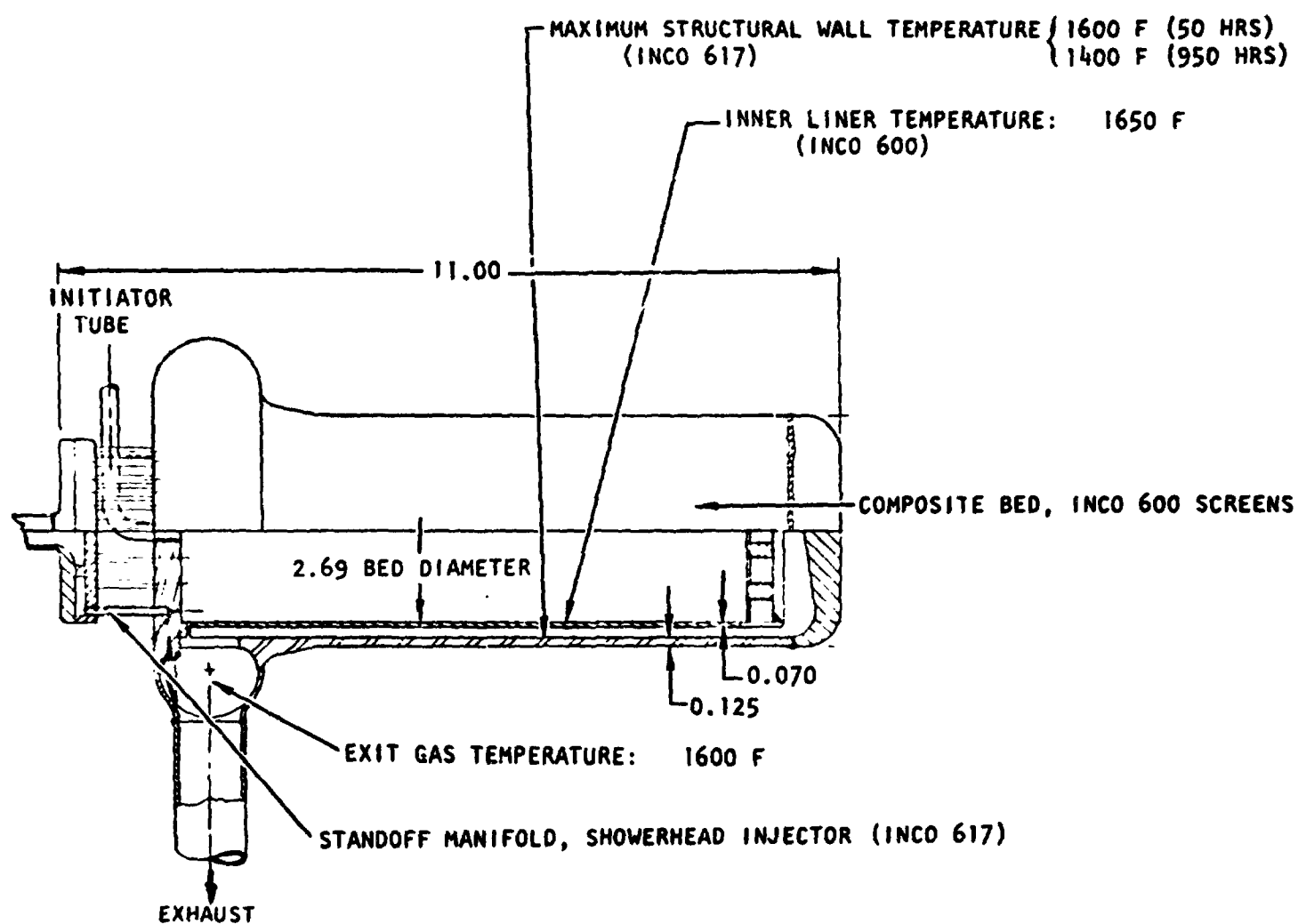


Figure 1. Features of Rocketdyne Design NAS9-13003

DISCUSSION

SELECTION OF CANDIDATE MATERIALS

Thermal distribution of the original gas generator design is shown in Fig. 2. The gas generator structural wall reached a maximum temperature of 1745 F and the thermal screens approximately 1850 F under steady-state maximum power conditions. Based on maximum steady-state operating pressures of 660 psia and transient pressures of 1000 psia, the required wall yield strength is 6800 psi and rupture strength is 4500 psi (using a safety factor of 1.5 and an 0.21-inch wall chamber). In selecting candidate materials it was assumed that the structural wall is exposed to a temperature of 1800 F for 100 hours, representing maximum power operation, and to a temperature of 1600 F for 900 hours, representing idle power operation.

The properties of various materials were scanned in an attempt to isolate those that combine high strength at 1800 F together with a low percentage of nitride-forming elements. Figure 3 presents the results of this analysis. The 0.2-percent yield strength is plotted in Fig. 3A, and Fig. 3B shows the 100-hour rupture strength as a function of total percentage of nitride formers.

Note that INCO 600 has the lowest percentage (16.5) of nitride formers (with the exception of the nonweldable TD nickel); however, its strength is low compared to the yield and rupture strength requirements stated above. On the other hand, while INCO 718, Hastelloy B, Rene 41, Hastelloy C, and INCO 625 have a yield strength above the 6800 psi requirement, their 100-hour rupture strength capability is near zero at 1800 F. On the basis of this comparison, candidate materials were selected for use in the evaluation program. The selected materials were divided into two groups: five prime candidates and four secondary candidates (Table 2). The candidate materials were machined into tensile specimens according to Rocketdyne specification TF-250.

100 PERCENT POWER LEVEL

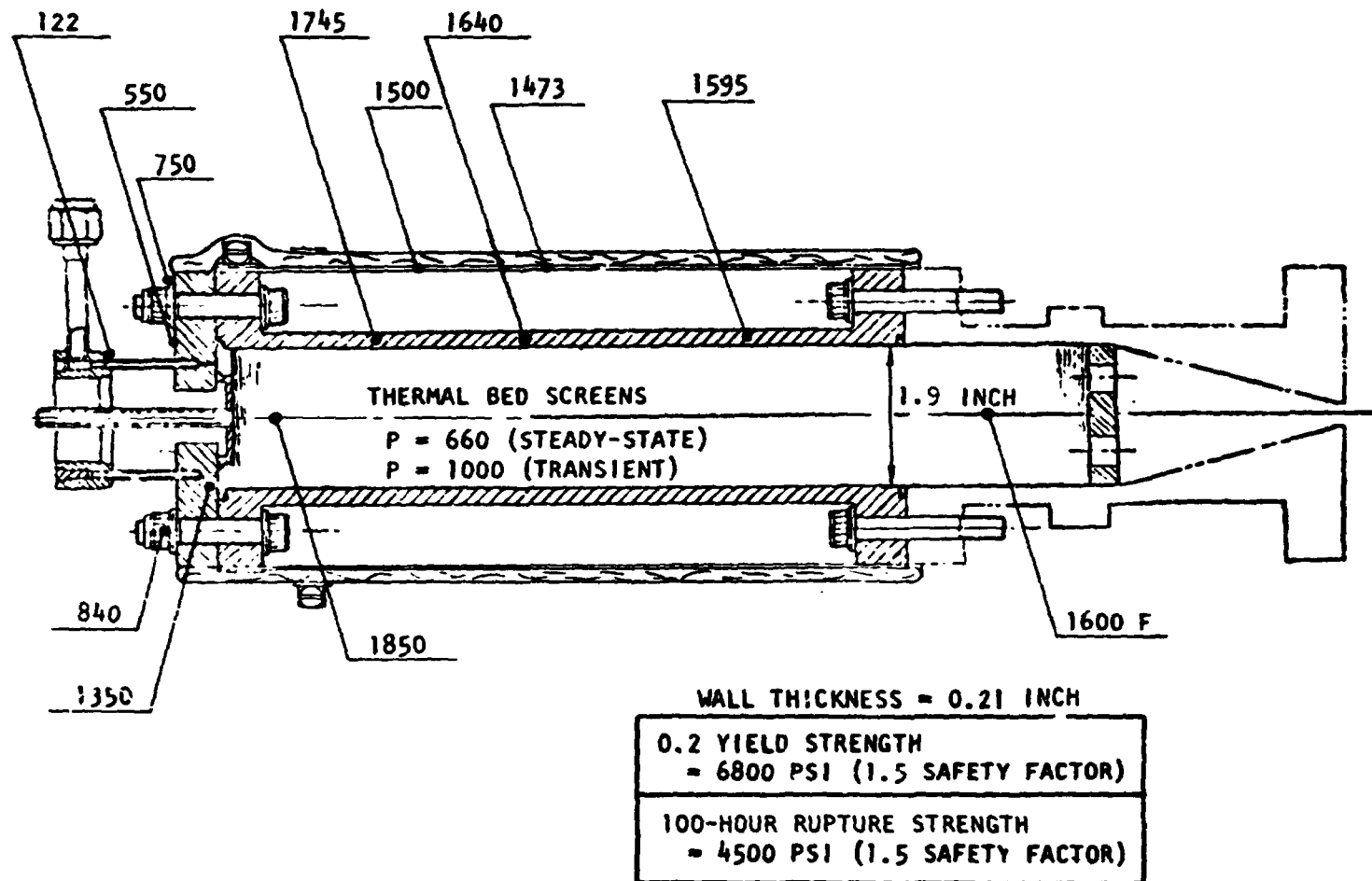


Figure 2. Flight-Type Gas Generator Temperature Distribution Test Data

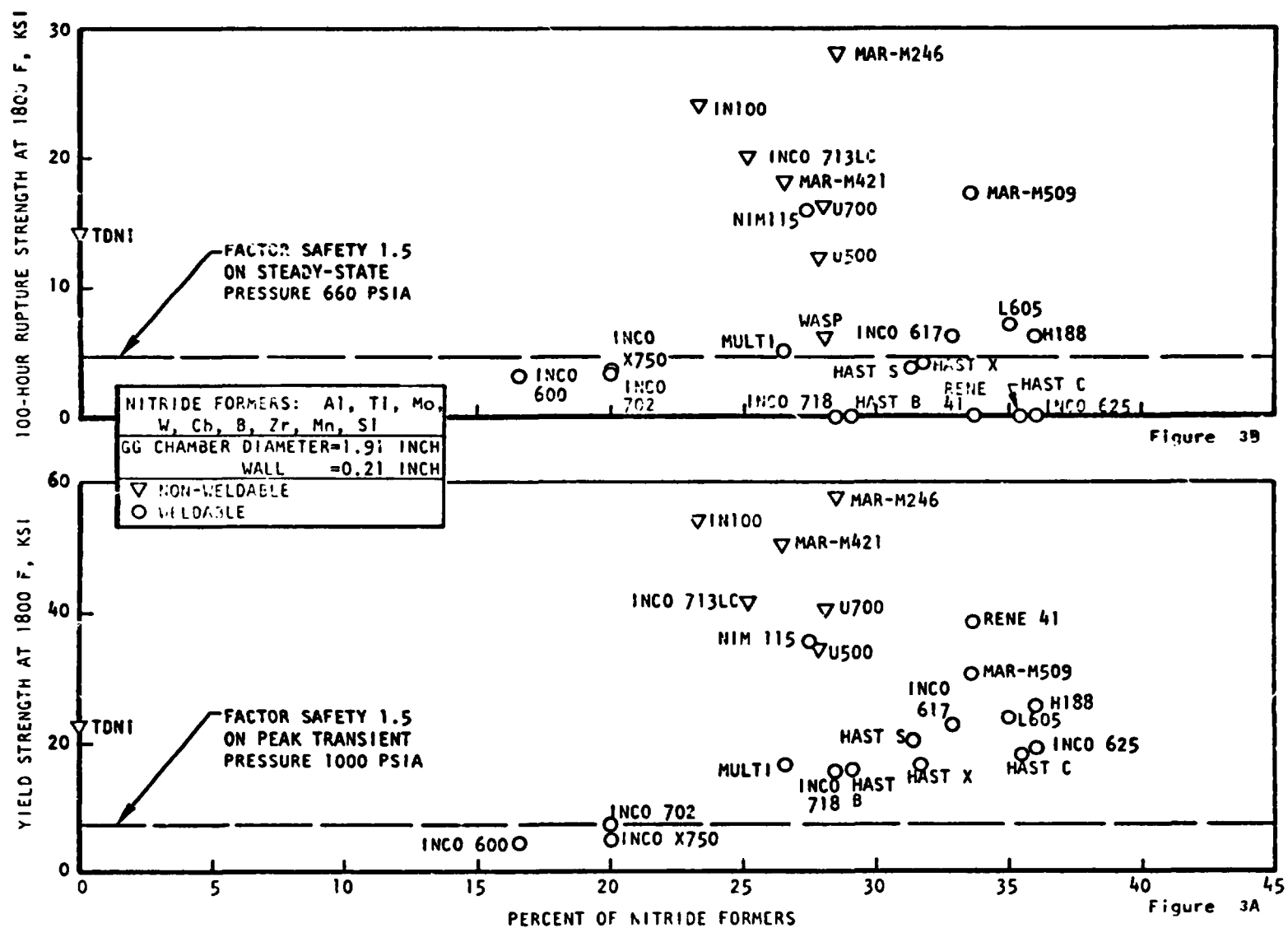


Figure 3. Comparison of Candidate Materials

TABLE 2. CANDIDATE MATERIALS

Prime Candidates	Secondary Candidates
L605	Multimet
Haynes 188	TD Nickel (nonweldable)
Inconel 617	MAR-M-509
Inconel 600	MAR-M-246 (nonweldable)
Nickel 270	

With the exception of the Nickel 270, the prime candidates all represent weldable alloys suitable for use as the gas generator structural wall. The INCO 600, being of lower strength, is not suitable for the structural wall unless the wall thickness is increased from 0.21 inch (Fig. 3). The Ni 270 and INCO 600 materials may be used as screen materials or in another low-strength application. The Ni 270, being void of nitride formers, also represents a control sample.

The secondary materials, which would undergo less extensive testing than the primary candidates, were estimated to have a lower probability of usefulness for the gas generator, due to fabrication considerations or percent of nitride formers.

Two chrome-plated INCO 617 tensile specimens (0.002-inch plate) were also included for the purpose of evaluating the resistivity of the plating to nitride penetration of the parent material. One of the plated specimens was oxidized in air for four hours at 1200 F before nitride exposure.

In addition to the tensile specimens, four individual INCO screens and two 1/2-inch-thick brazed screen packs were included in the 1000-hour test. The screens were all constructed of 20 mesh \times 0.025-inch-diameter wire. The screen pack was brazed around the outer cylindrical surface with a Palniro No. 4 braze consisting of 30-percent gold, 34-percent platinum, and 36-percent nickel. The braze alloy melting point is 2136 F, but after alloying with the INCO 600 screen material the probable melting temperature is in excess of 2200 F.

A photograph of the material tensile specimens and brazed screen pack is shown in Fig. 4 .

TEST PROGRAM

All the tensile specimens and screens were installed in a 2-1/4-inch diameter, 18-inch long cylinder through which 1800 F NH_3 gas, at atmospheric pressure, was introduced. As a result of dissociation of the NH_3 gas passing through the cylinder, an 1800 F gas generator environment with 60 percent NH_3 dissociation was approximated. All material samples were cleaned, weighed, and measured before installation in the oven. Four samples each of the primary candidate tensile specimens, three samples each of the secondary candidate tensile specimens, and two samples of the chrome-plated INCO 617 specimens were fabricated. The test program is outlined in Table 3 .

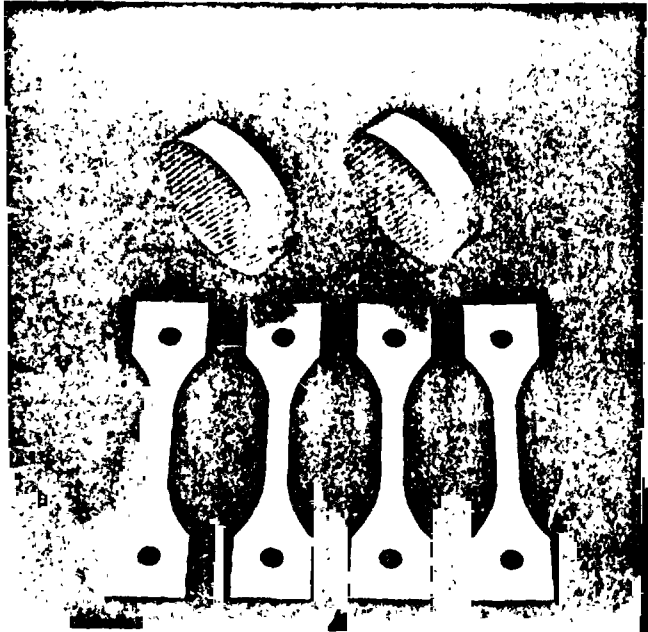
TEST RESULTS

Tensile specimens were weighed and their thickness measured before and after exposure to the 1800 F nitriding environment. These data are shown in Appendix A. The data for samples 2 and 3 (10 hours and 100 hours) of the primary candidate specimens are shown on page 40 ; sample 3 (100 hours) data of the secondary candidate specimens are shown on page 41 ; and sample 4 (1000 hours) data of both the primary and secondary candidates on pages 42 and 43, respectively.

High-temperature (1800 F) tensile test data of the specimens are presented in Appendix B. Sample 1 (unexposed), sample 2 (10 hours), and sample 3 (100 hours) of the primary candidate specimens are shown on pages 46, 47, and 48 , respectively. Sample 1 (unexposed) and sample 3 (100 hours) of the secondary candidate specimens are shown on page 49. Sample 4 of both the primary and secondary candidate specimens is shown on page 50.

All tensile testing was conducted at 1800 F with an argon atmosphere protecting the specimen during the entire test cycle. Heat-up time was approximately

MATERIAL SPECIMENS



MATERIAL NITRIDING TEST

1800 F NITRIDING ENVIRONMENT
EXPOSURE FOR 10, 100, 1000 HRS

- TENSILE SPECIMENS
- THERMAL BED SCREENS
- BRAZED SCREEN PACK

INCO 600
INCO 617
HAYNES 188
L605
Ni 270

MULTIMET
MAR M-509
MAR M-246
TD Ni

Figure 4. 1000-Hours Material Nitriding Test Program

TABLE 3. TEST PROGRAM

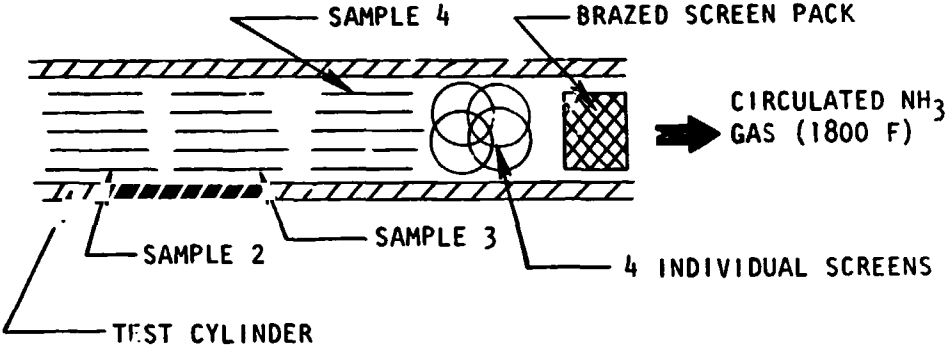
Time	Action
0	<ol style="list-style-type: none"> 1. Weigh and measure four samples each of the primary candidate (PC) specimens. 2. Install samples 2, 3, and 4 of the PC tensile specimens into the oven. 3. Tensile test sample 1 of PC specimens (unexposed). 4. Install INCO 600 screens (4) and one of the brazed screen packs into the oven. 5. A schematic of the oven test setup is shown below:  <p>The diagram shows a cross-section of a 'TEST CYLINDER' represented by two parallel horizontal lines. Inside the cylinder, from left to right, are 'SAMPLE 2', 'SAMPLE 3', and 'SAMPLE 4'. Between these samples are '4 INDIVIDUAL SCREENS', depicted as small squares with diagonal lines. To the right of the cylinder is a 'BRAZED SCREEN PACK', shown as a larger square with a cross-hatch pattern. An arrow points from the right towards the cylinder, labeled 'CIRCULATED NH₃ GAS (1800 F)'.</p>
10 Hours	<ol style="list-style-type: none"> 1. Remove sample No. 2 from oven. Move samples No. 3 and 4 upstream so that sample 3 is in the former position of sample 2. 2. Measure, weigh, and tensile test sample 2 of PC specimens. 3. Remove screen pack from oven, vibrate on shake table, and replace in oven. Vibration characteristics were as follows: <ul style="list-style-type: none"> Maximum Frequency = 120 Hz Maximum Amplitude 0.062 inch Peak Acceleration = 20 to 30 g at 120 Hz

TABLE 3. (Continued)

Time	Action
100 Hours	<ol style="list-style-type: none"> 1. Remove sample 3 of PC specimens from oven. Move sample 4 upstream to the former position of sample 3. 2. Measure, weigh, and tensile test sample 3 of PC specimens after 100 hours exposure. 3. Remove screen pack from oven, vibrate on shake table, and replace.
603 Hours	<ol style="list-style-type: none"> 1. Weigh and measure three samples each of secondary candidate specimens and the two chrome-plated INCO 617 specimens. 2. Install samples 3 and 4 of the secondary candidate (SC) tensile specimens into the oven together with the two chrome-plated specimens. 3. Tensile test sample 1 of the SC specimens. 4. A schematic of the oven test setup at this time is shown below. <div data-bbox="469 1228 1401 1618"> <p>The diagram illustrates the oven test setup at 603 hours. It shows a cross-section of a furnace with two horizontal specimen holders. The top holder contains 'SAMPLE 4 (SC SPECIMENS) + 2 CHROME-PLATED SPECIMENS' and '4 INDIVIDUAL SCREENS'. The bottom holder contains 'SAMPLE 3 (SC SPECIMENS)' and 'SAMPLE 4 (PC SPECIMENS)'. A 'BRAZED SCREEN PACK' is shown to the right of the specimen holders. An arrow points from the left towards the specimen holders.</p> </div>

TABLE 3. (Concluded)

Time	Action
703 Hours	<ol style="list-style-type: none"> 1. Remove sample 3 of SC specimens from oven; measure and weigh. 2. Tensile test sample 3 of SC specimens after 100-hour exposure
1000 Hours	<ol style="list-style-type: none"> 1. Remove all material specimens from oven; measure and weigh. 2. Tensile test sample 4 of PC specimens, and sample 4 of SC specimens. 3. Conduct hardness penetration test on samples 1, 2, and 3 (0-, 10-, and 100-hour exposure) of the INCO 600, L605, and Haynes 188 specimens. 4. Conduct a room temperature bend test on the sample 4 tensile specimens after tensile testing. 5. Conduct a room temperature bend test on the 1000-hour exposed screens. 6. Take micrographs of tensile specimens after various exposure times. 7. Analyze low-cycle fatigue characteristics of the Haynes 188, INCO 600, and L605 based on the tensile test data.

1 hour and 20 minutes, plus 10 minutes for stabilizing at the test temperature, plus 5 minutes for the test. The total exposure of each tensile specimen to high temperatures (over 1600 F) in argon was approximately 55 minutes.

Figures 5 through 13 present results of the tensile testing and the weight and thickness measurements for all the specimens. The 1000-hour strength data for Ni 270 (Fig. 8) was not available because of damage to the tensile specimens following the oven test. Also, due to shortage of L605 material, no sample 4 test specimen was available for the 1000-hour test data (Fig. 9).

In general, the tensile specimens experienced either little change or an increase in both yield and ultimate strength. Percent elongation and reduction of area percentages were significantly reduced with exposure time, with the exception of Multimet and TD Nickel. Multimet, which recorded an increase in percent elongation after 400 hours exposure, indicated extremely low room temperature ductility; the 100-hour tensile specimen snapped when accidentally tapped with a tool. TD Nickel, which also recorded an increase in percent elongation after 400 hours exposure, had substantially lower strength (less than 5000 psi yield) than expected. Of the remaining high-strength materials (yield strength in excess of 15,000 psi), INCO 617 demonstrated the highest percent elongation after 1000 hours exposure, and L605 the highest after 100 hours exposure.

The materials showed either a small change or an increase in surface hardness after 400 hours exposure, with the exception of MAR-M-246 (an unweldable cast alloy), TD Nickel, and INCO 600.

The two chromeplated INCO 617 specimens lost their coatings during the 100-hour exposure period. The 1/2-inch Inconel 600 screen pack showed a 1.96-percent increase in weight and a 5.13-percent increase in length after 1000 hours. The screen pack was removed from the oven after 10 hours and 100 hours and was vibrated on a shake table. After 1000 hours, the braze was intact and appeared satisfactory for use in a gas generator bed.

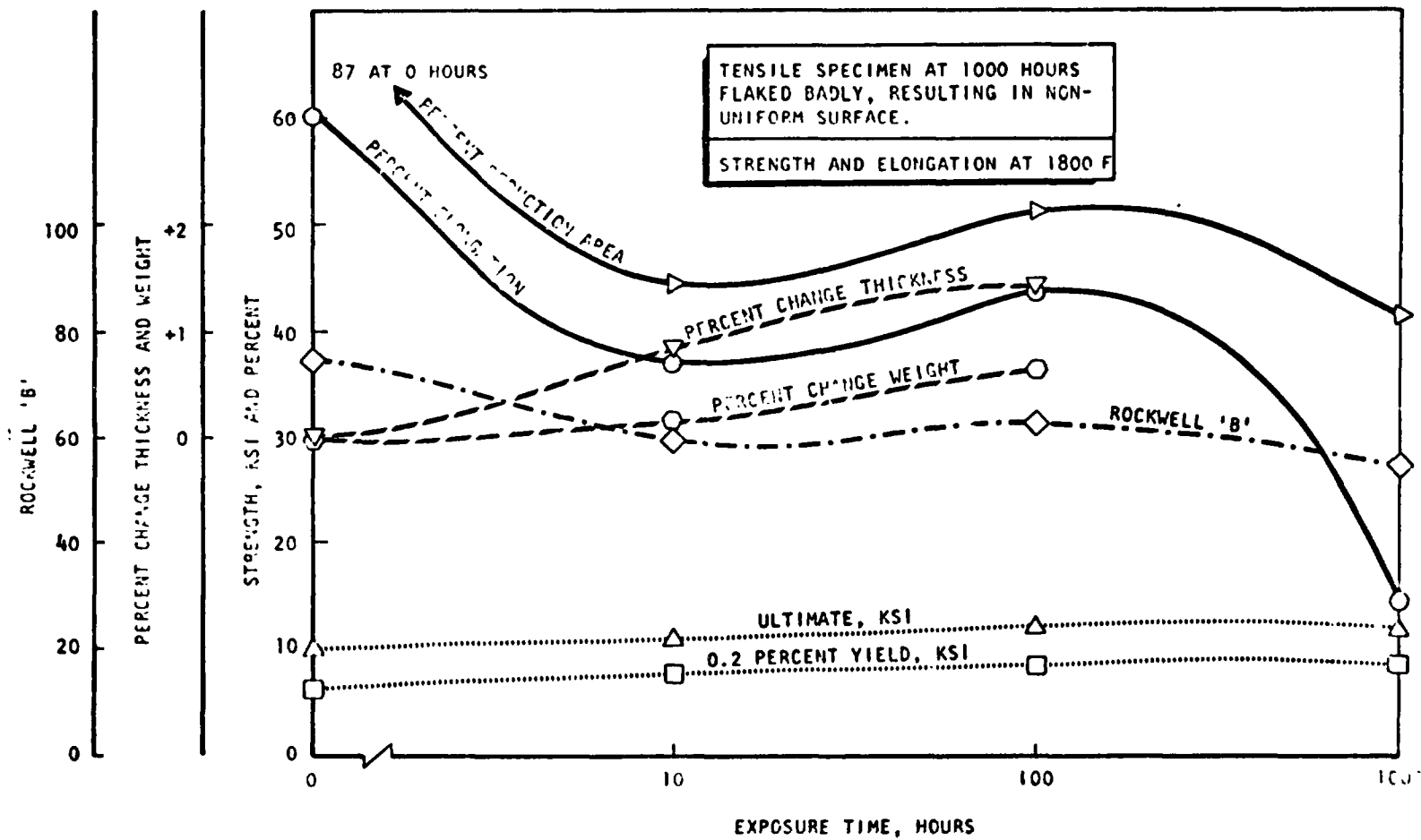


Figure 5. Material Nitriding Experiment - INCO 600

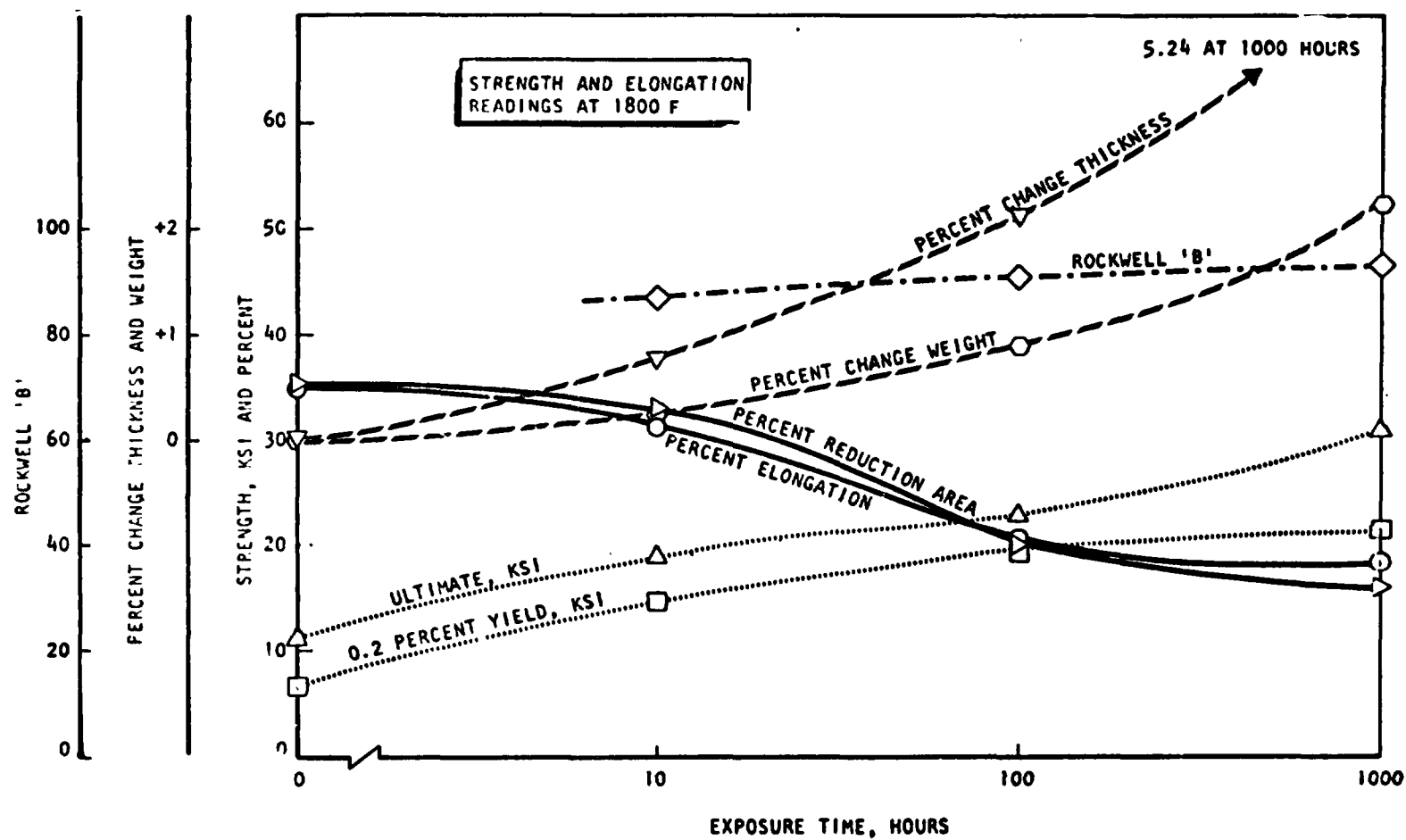


Figure 6. Material Nitriding Experiment - INCO 617

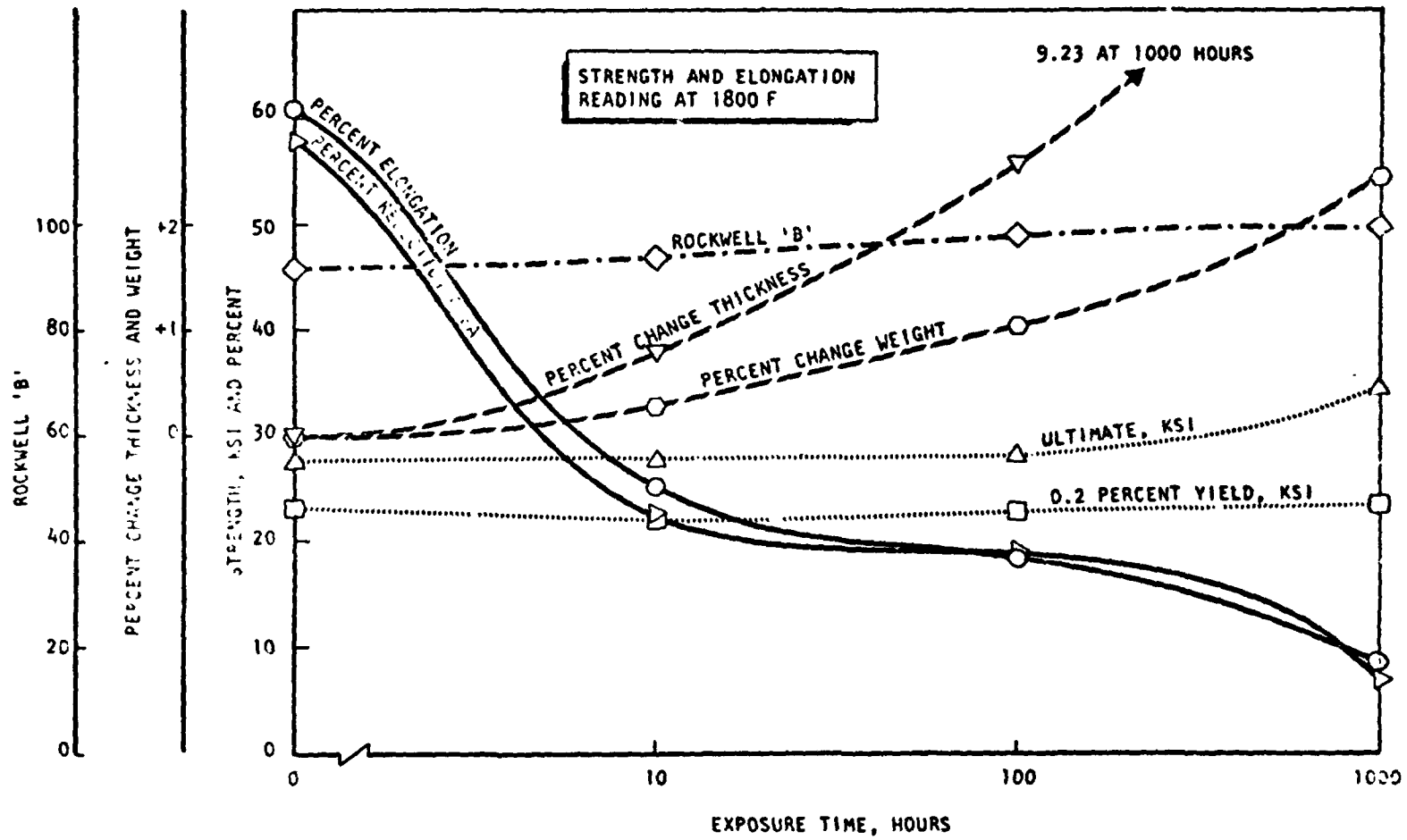


Figure 7. Material Nitriding Experiment - Haynes 188

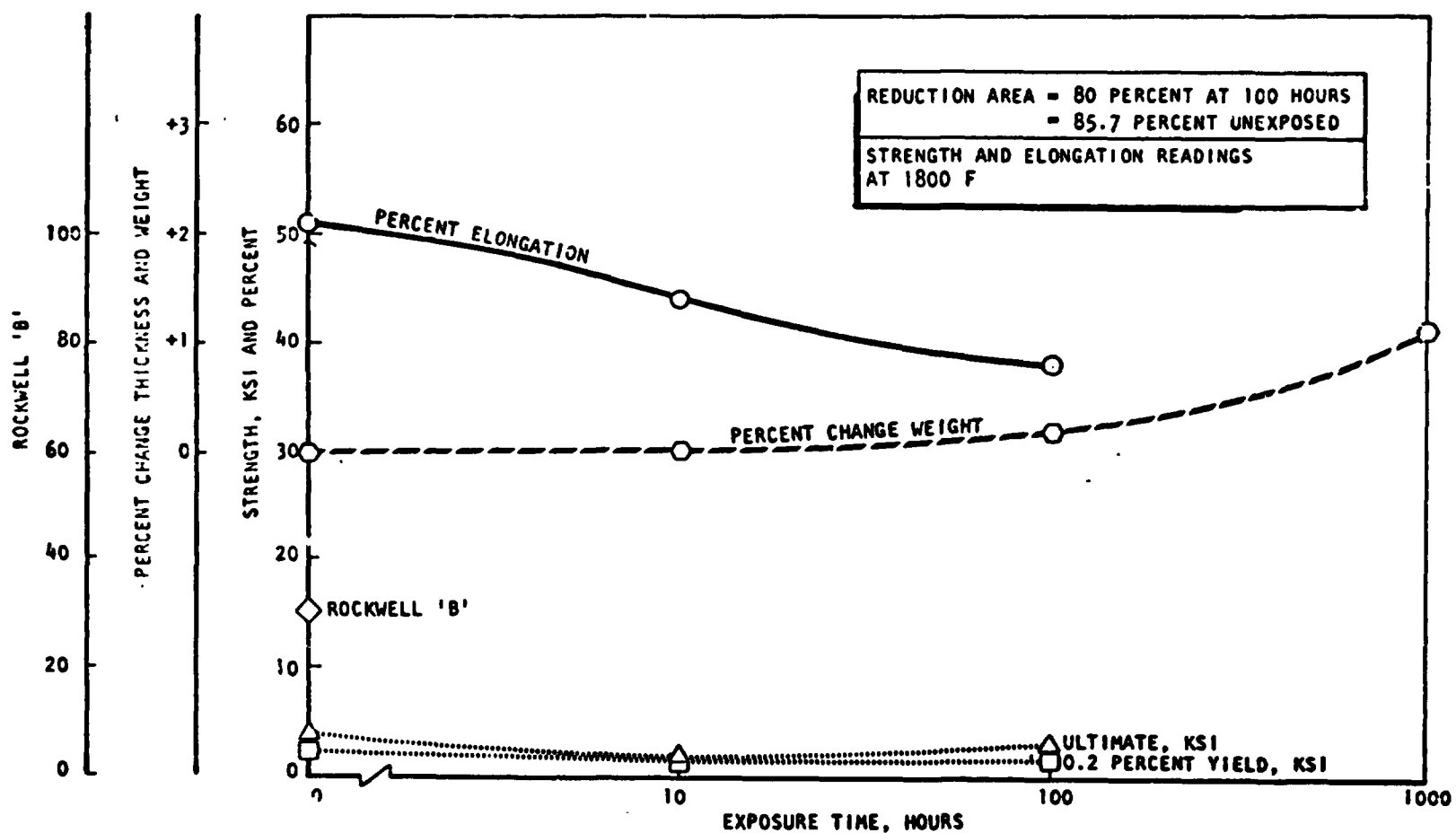


Figure 8. Material Nitriding Experiment - Ni 270

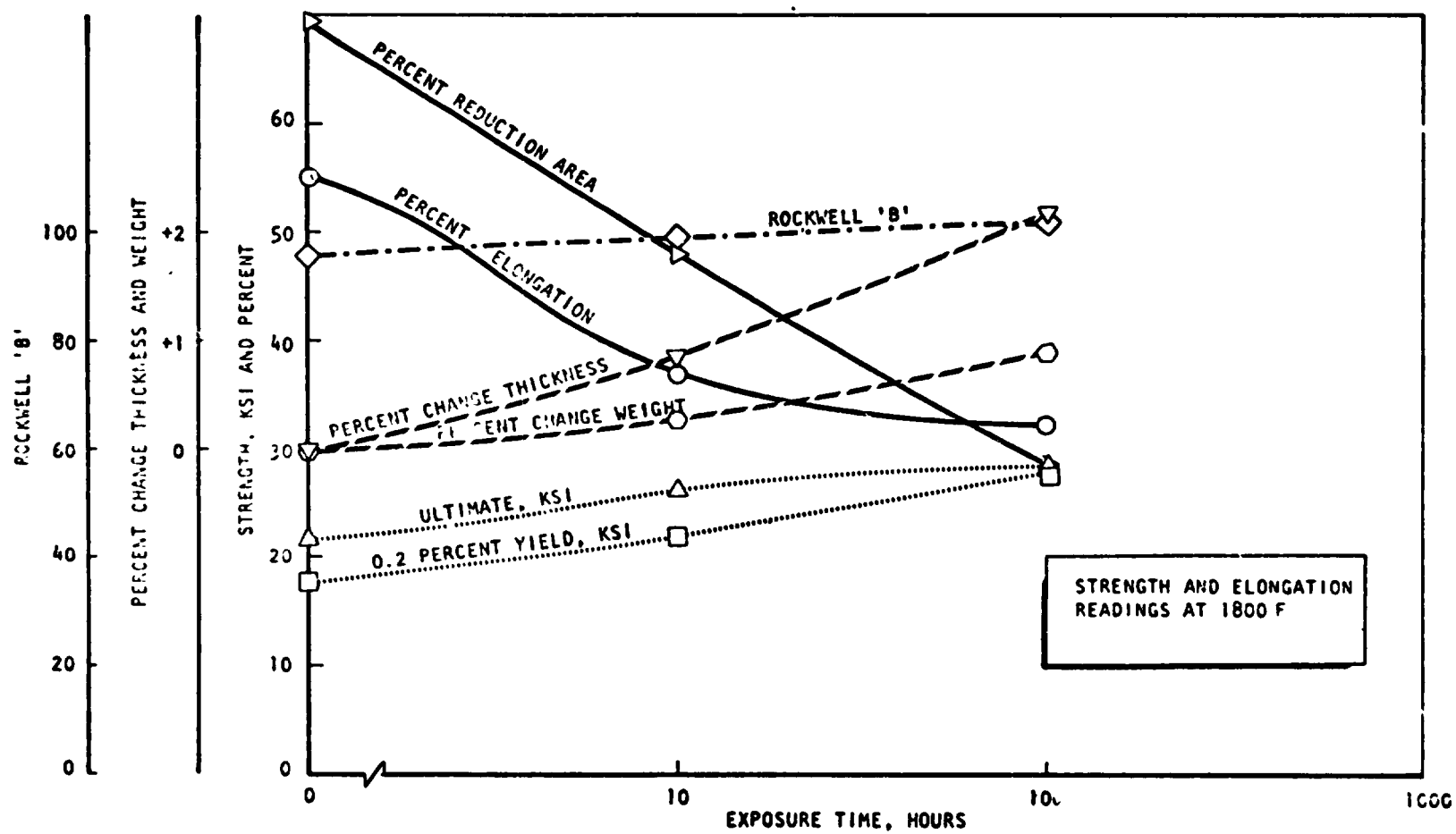


Figure 9. Material Nitriding Study - L605

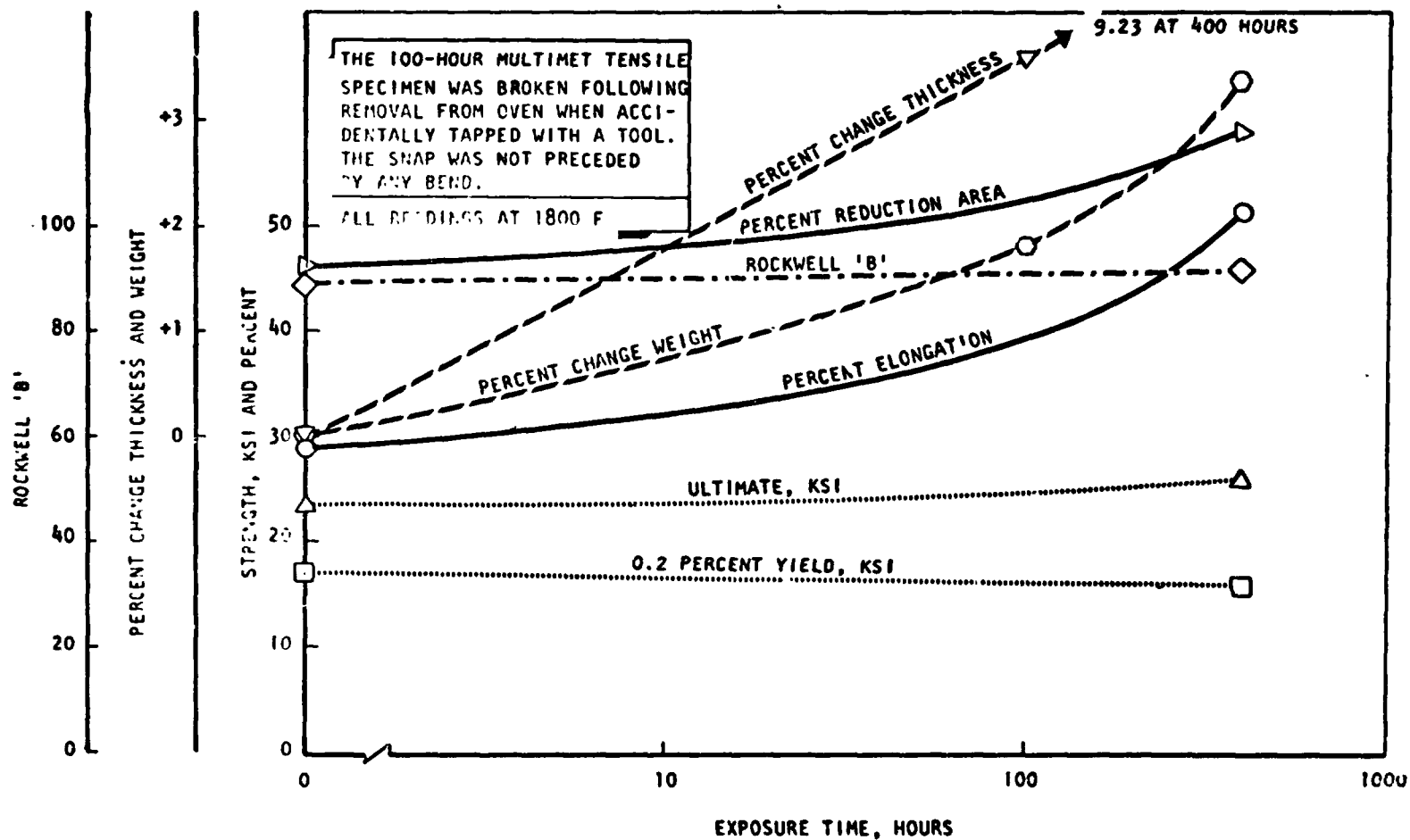


Figure 10. Material Nitriding Experiment - Multimet

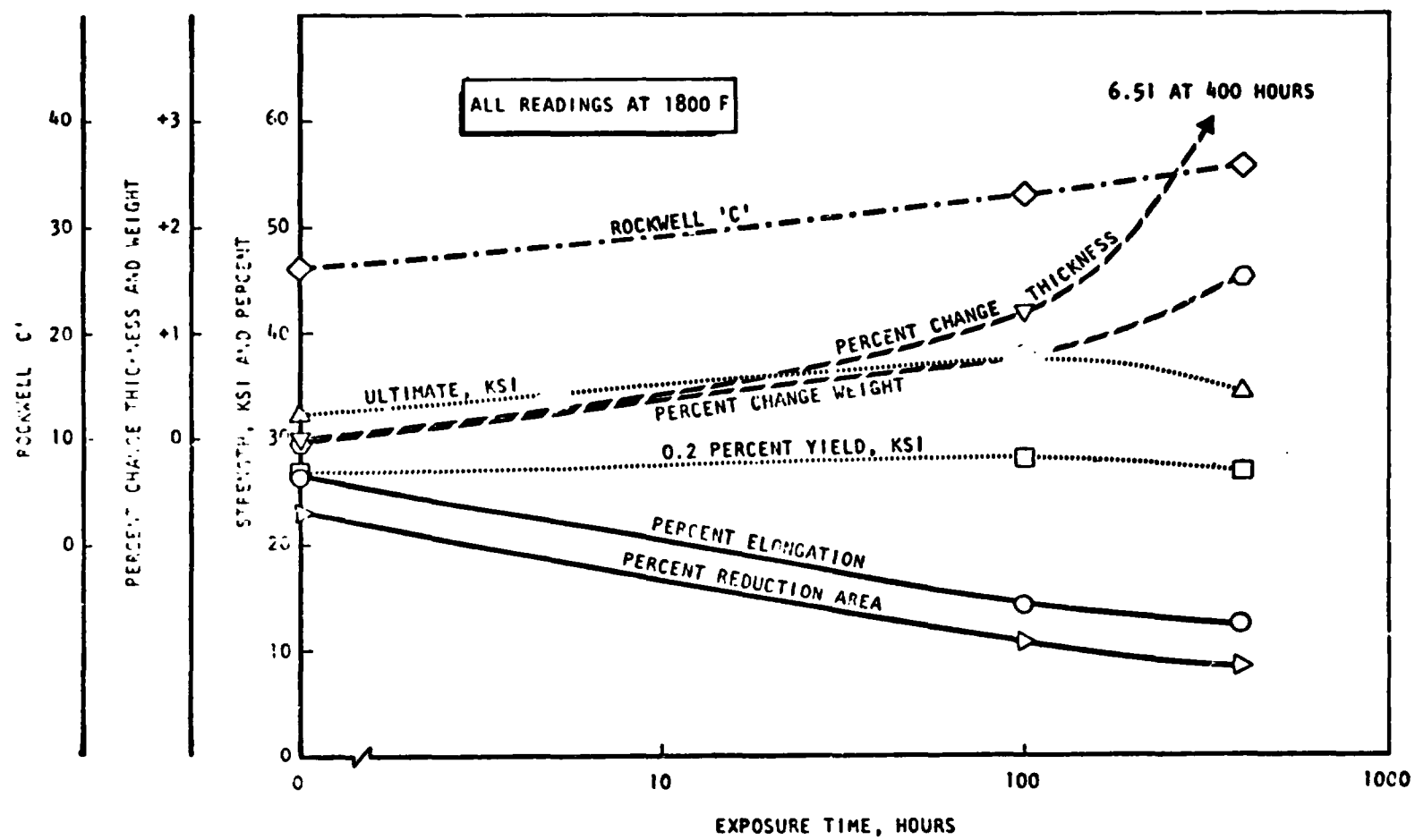


Figure 11. Material Nitriding Experiment - MAR-M-509, (Cast Alloy)

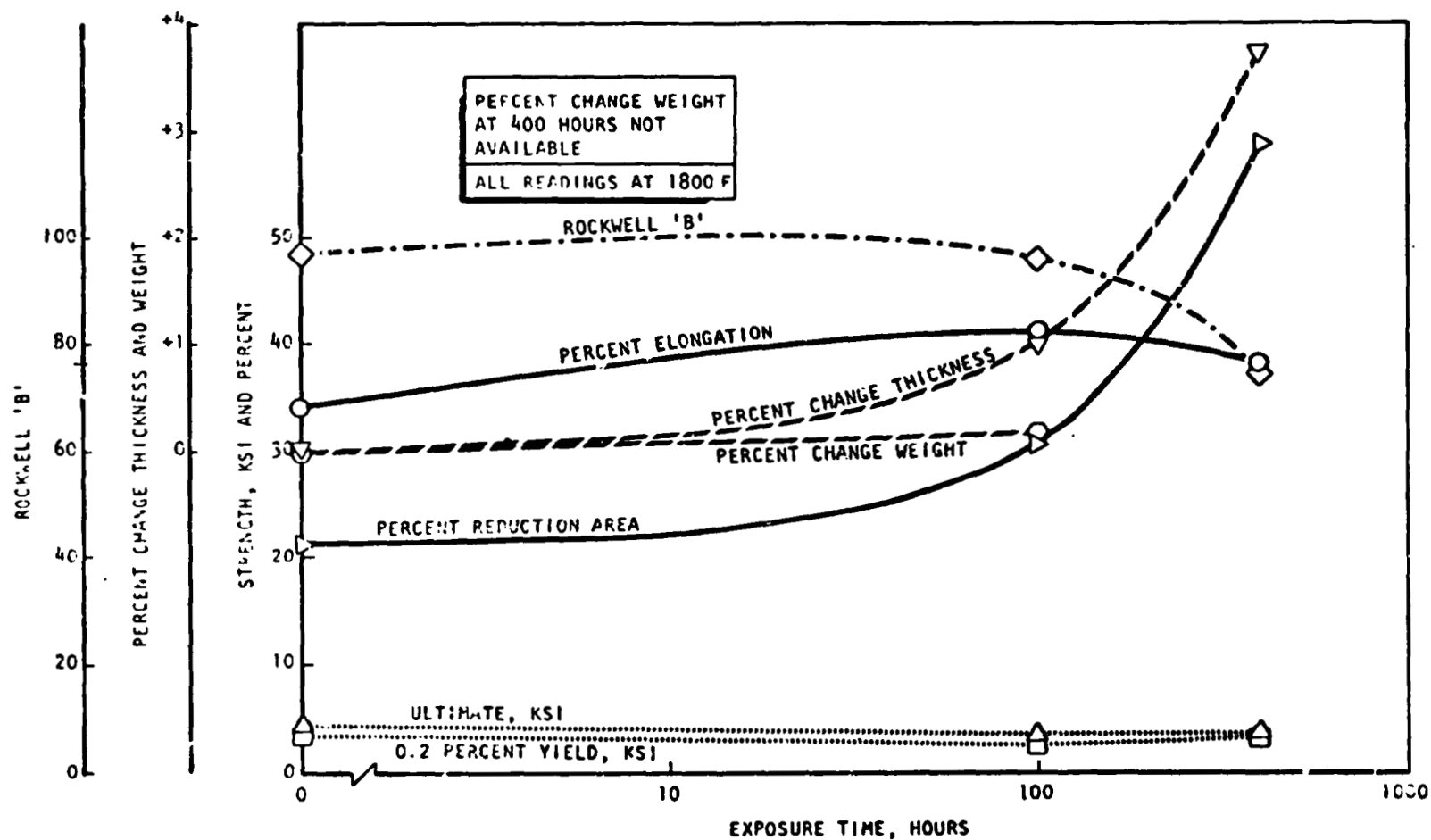


Figure 12. Material Nitriding Study - TD Nickel

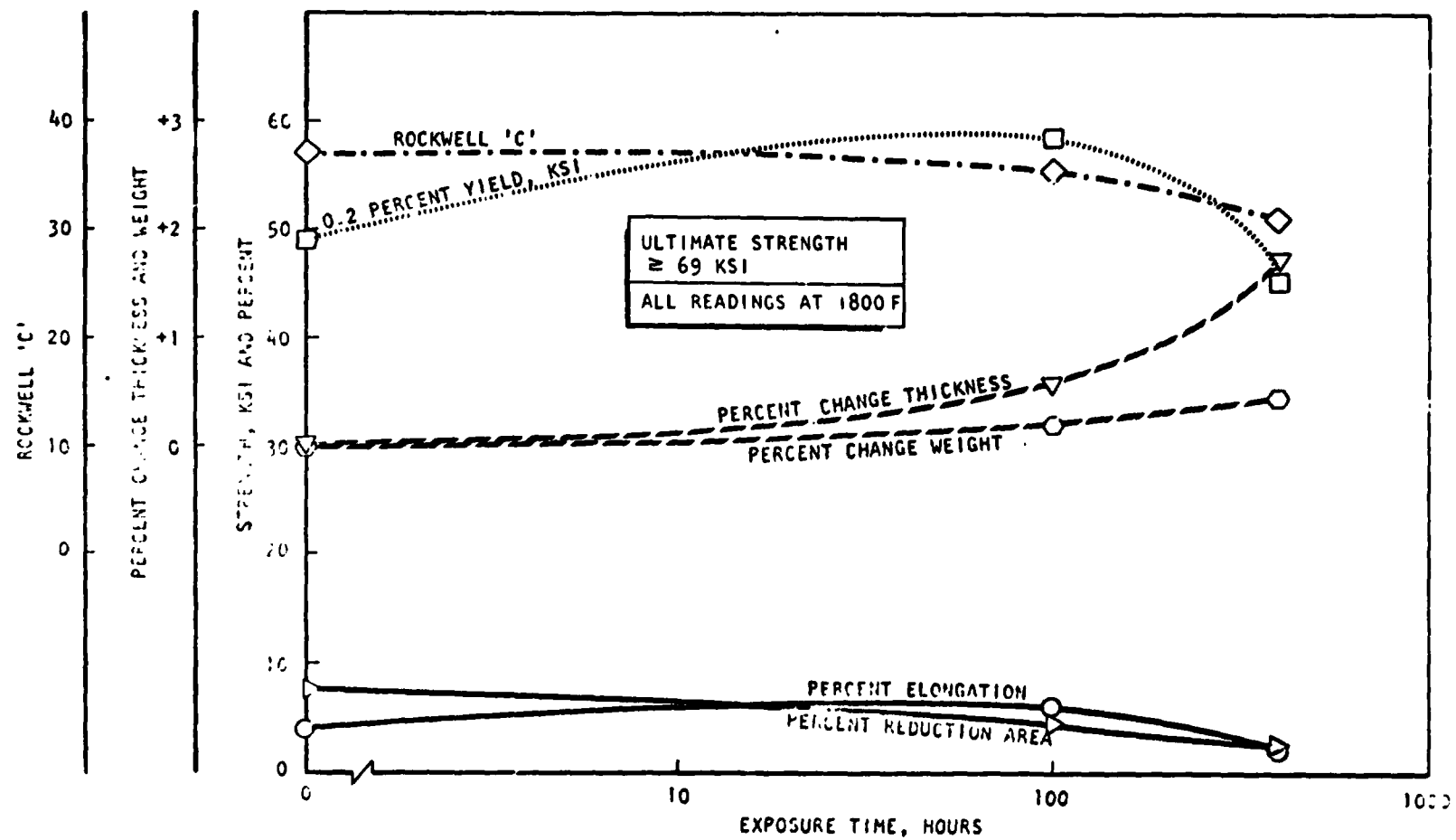


Figure 13. Material Nitriding Study - MAR-M-246, (Nonweldable, Cast Alloy)

Transverse hardness tests were conducted on the INCO 600, L605, and Haynes 188 specimens following tensile tests of the 0-, 10-, and 100-hour samples to evaluate both the nitriding rate and effect on hardness. The data are presented on pages 52 through 54 of Appendix C. Figures 14 through 16 present the results of these tests. In the construction of these curves, inverse hardness (filair unit penetration) is plotted as a function of distance from the tensile specimen surface. For example, in Fig. 14, the 0-hour sample had a relatively constant hardness across the 0.049-inch-thick specimen, being only slightly harder at the surface; after 10 hours of exposure, the hardness increased at the surface, but was unaffected beyond a depth of 0.010 inch.

After 100 hours of exposure, the INCO 600 indicated the least increase in hardness for depths greater than 0.010 inch. Surface hardness for the 100-hour INCO 600 specimen was also minimum. Of the two high-strength materials (L605 and Haynes 188), the Haynes 188 indicated least increase in hardness beyond 0.010 inch.

Bend tests were made on sample 4 (maximum exposure time) of the tensile test specimens and the individual screens (Fig. 17 and 18). After an 180-degree bend, the screen was inspected under a microscope. No cracks of the wire were evident.

Results of the tensile specimen bend testing are shown in Table 4. A slight crack was evident after the INCO 600, 1000-hour exposure, specimen was bent 180 degrees. This represents the highest room temperature ductility of all the test specimens examined. Of the high-yield strength materials, INCO 617 experienced the highest room temperature ductility after 400 hours of exposure. Even the INCO 617, however, indicated a substantial loss in ductility. The 0-hour control sample was bent 180 degrees with no cracks; whereas, after 1000 hours, a break occurred at a 25- to 30-degree bend angle. Metallographic specimens of the INCO 617, INCO 600, and Haynes 188 materials were prepared from the tensile test bars and are shown in Fig. 19 and 20. These specimens show the effect of the nitriding environment on the grain structure as a function of time. Figure 19 illustrates that the INCO 600 grain structure was affected for a depth of at least 0.005 inch after 100 hours

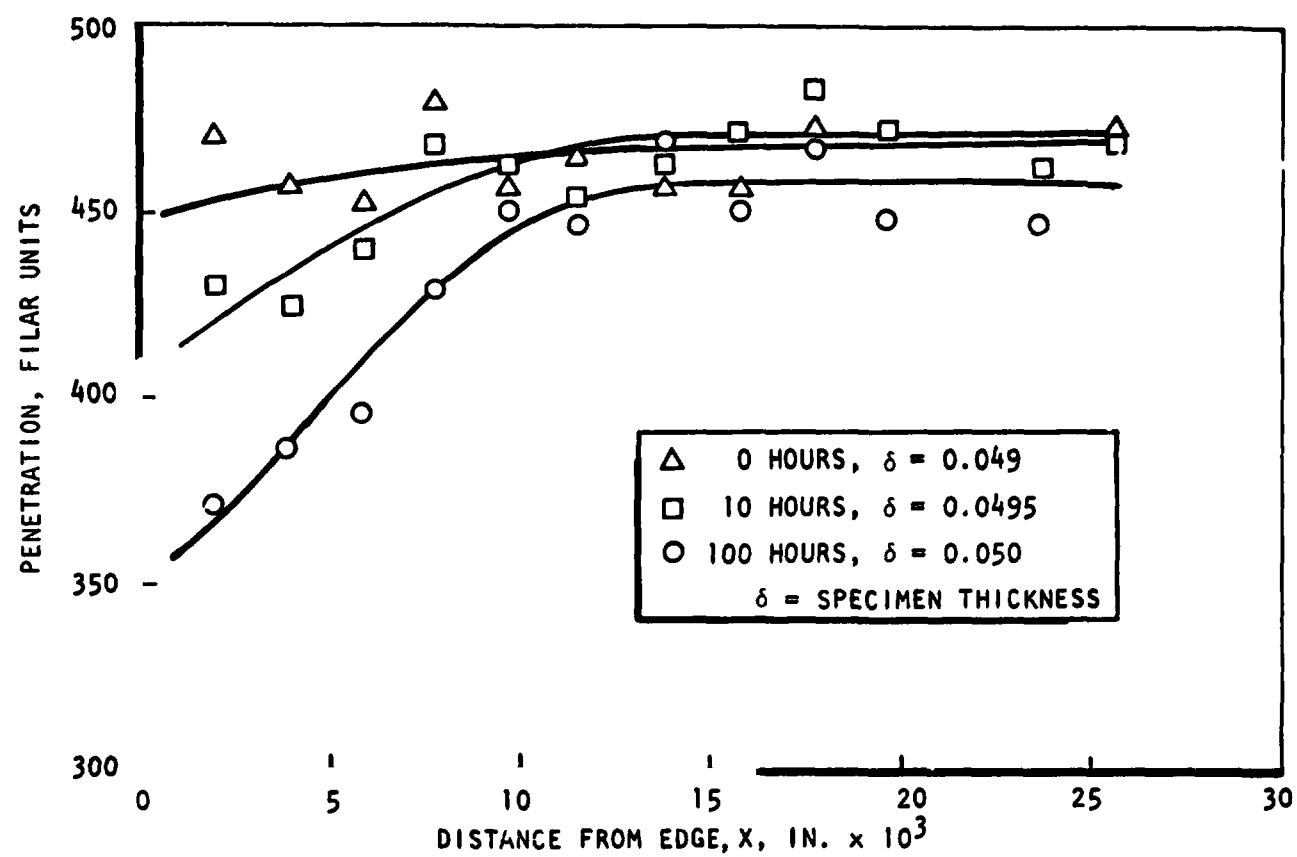


Figure 14. Material Nitriding Experiment Transverse Hardness - INCO 600 Tensile Specimen, (Exposed Both Sides)

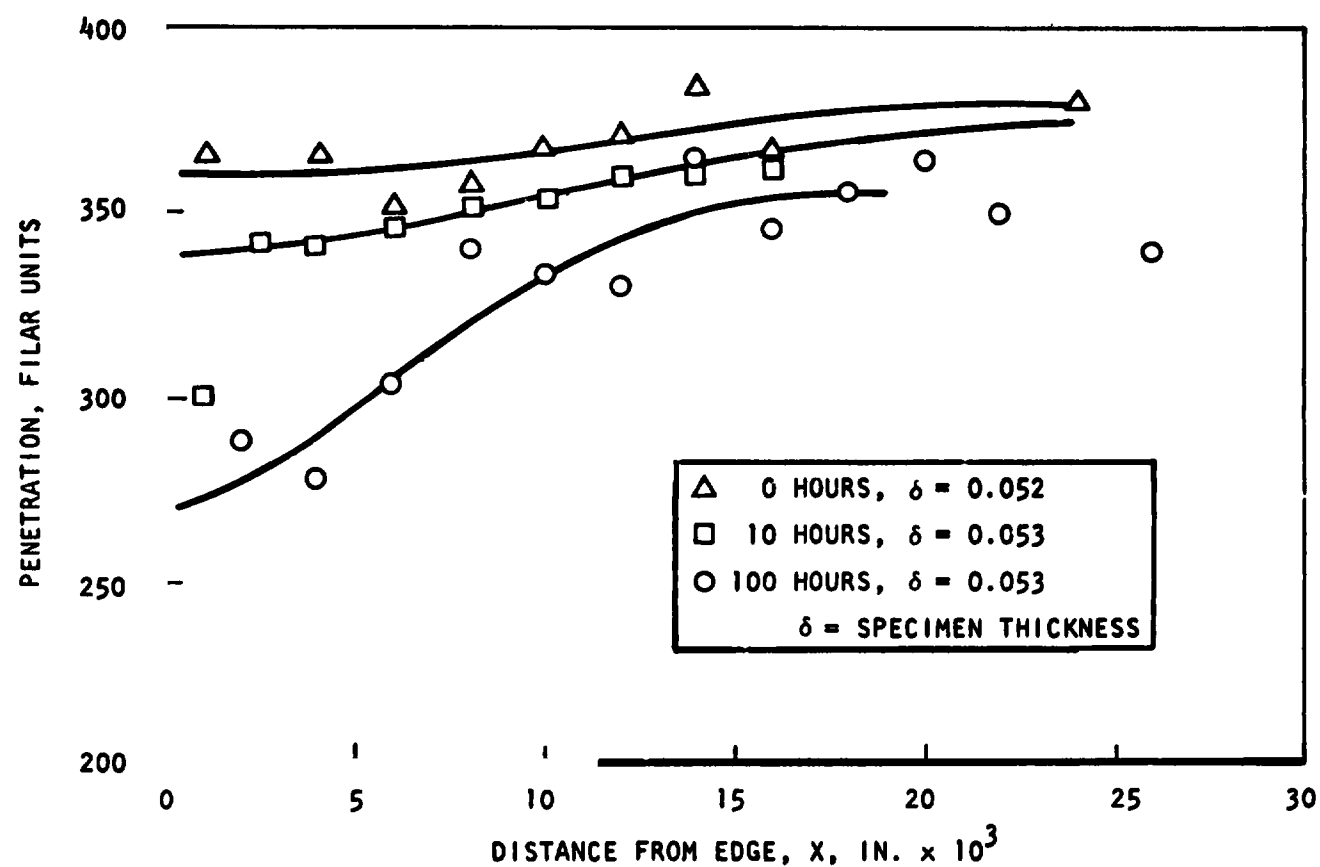


Figure 15. Material Nitriding Experiment Transverse Hardness - Haynes 188 Tensile Specimen, (Exposed Both Sides)

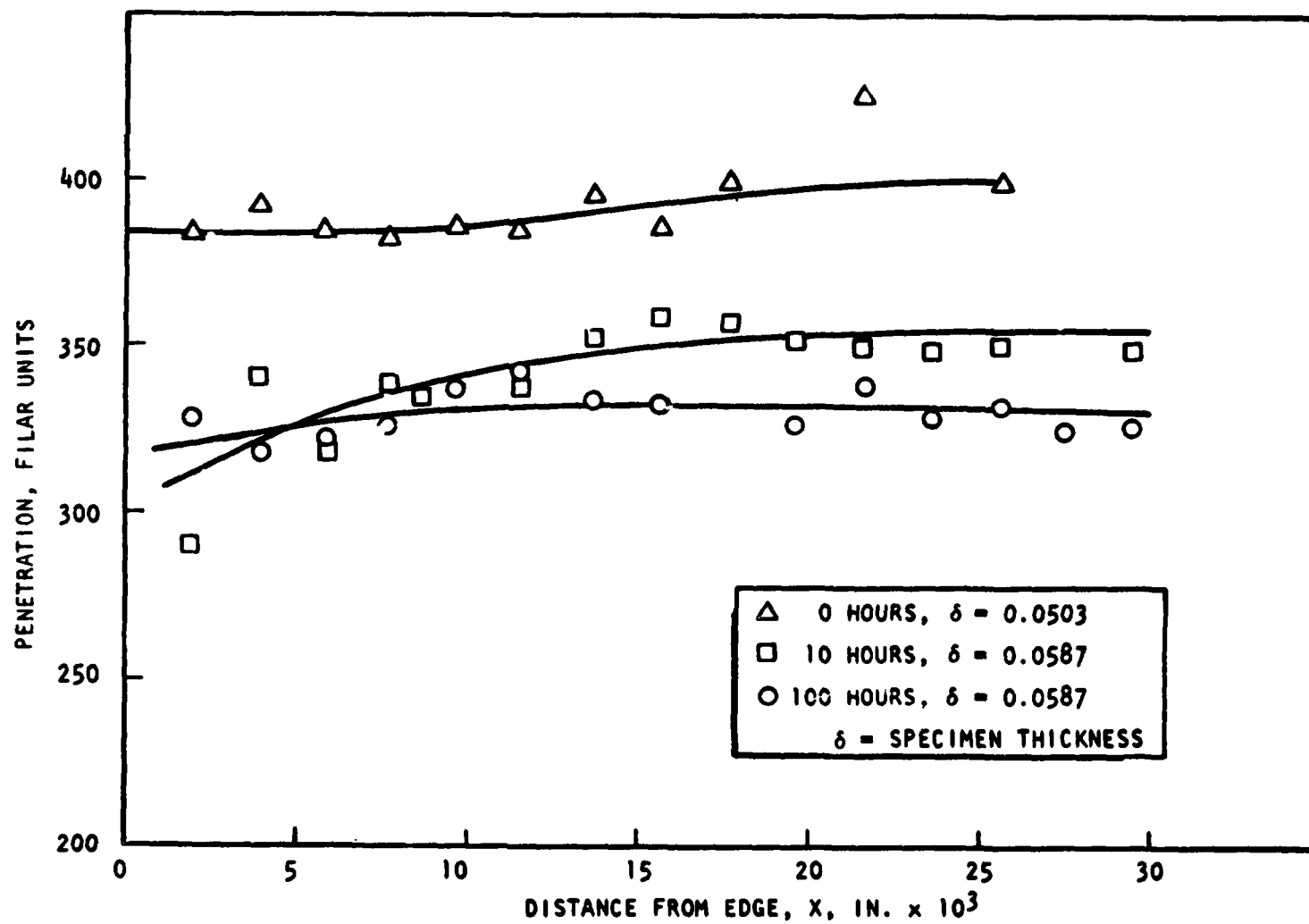


Figure 16. Material Nitriding Experiment Transverse Hardness-
L605 Tensile Specimen, (Exposed Both Sides)

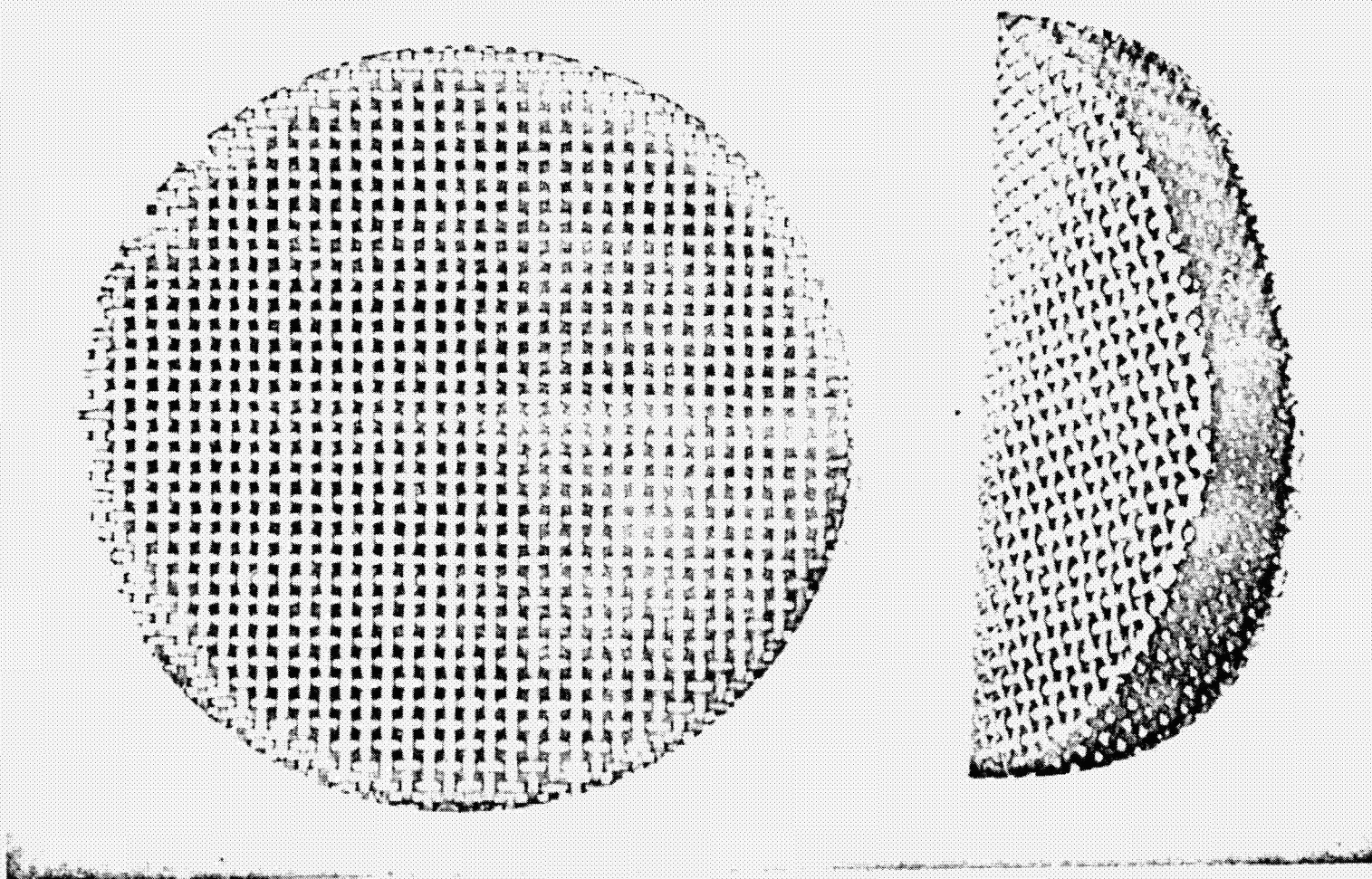


Figure 17. INCO 600 Screen 1000-Hours Exposure Bend Test

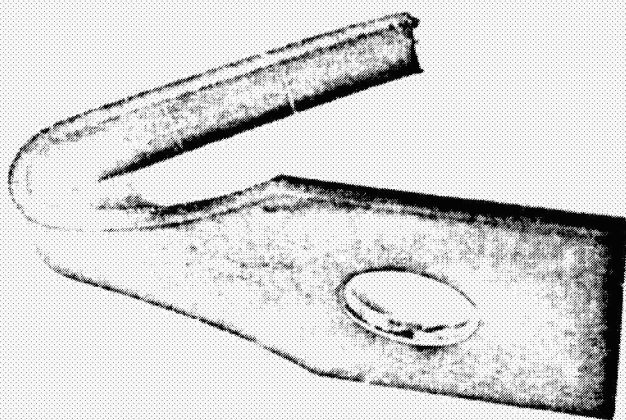
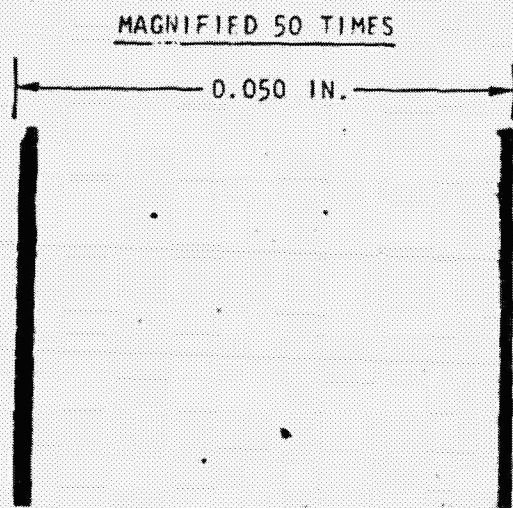


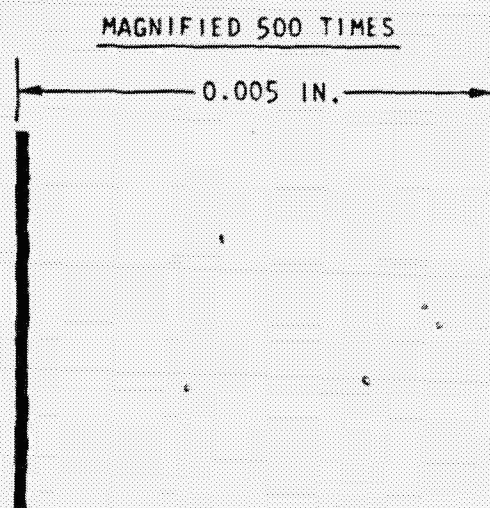
Figure 18. INCO 617 10-Hour Tensile Specimen Bend Test

TABLE 4. TENSILE SPECIMEN INSPECTION AND BEND TEST

Material	Sample (Exposure Time)	Bend Angle, degrees	Visual Inspection of Tensile Specimen
INCO 600	4 (1000 hours)	180 (slight crack)	Brittle dark scale. Base material ductile.
Multimet	4 (397 hours)	0	Brittle dark scale, 0.003-inch thick, peeling. Brighter material below scale is also cracked in reduced section of specimen.
INCO 617	4 (1000 hours)	25 to 30 (broke)	Sample 4: dark scale-like coating, 0.0015-thick. Small cracks near edge of bar, and near fracture on the flat surface.
	3 (100 hours)	45 (cracked)	
	2 (10 hours)	180 (no crack)	
	1 (0 hours)	180 (no crack)	
Haynes 188	4 (1000 hours)	0	Dark scale-like coating (0.003-inch-thick) all over, but not flaking off.
TD Ni	4 (397 hours)	180 (no crack)	Bright surface. No scale. Surface is cracked near fracture.
MAR M-509	4 (397 hours)	0	Blue-gray scale which is cracked only near fracture. Coating thickness is 0.0015 inch.
MAR M-246	4 (397 hours)	10 to 15	Dark surface, extremely thin scale thickness with cracking.



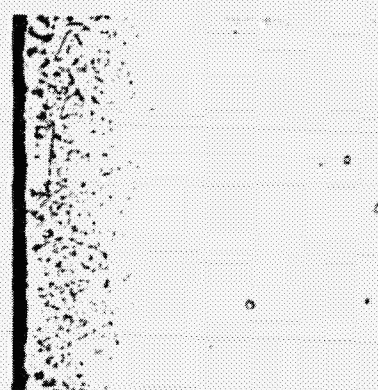
0-HOUR EXPOSURE



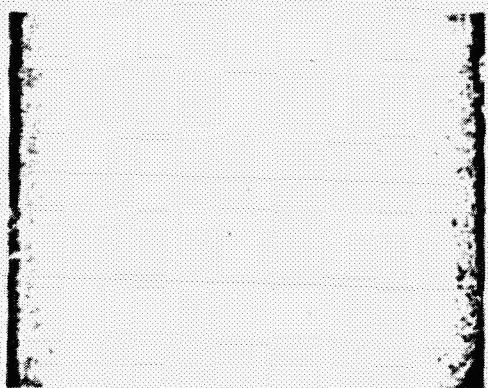
0-HOUR EXPOSURE



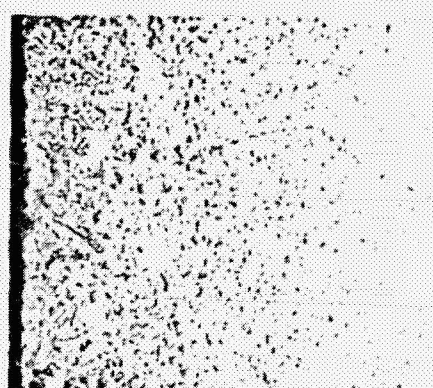
10-HOURS EXPOSURE



10-HOURS EXPOSURE

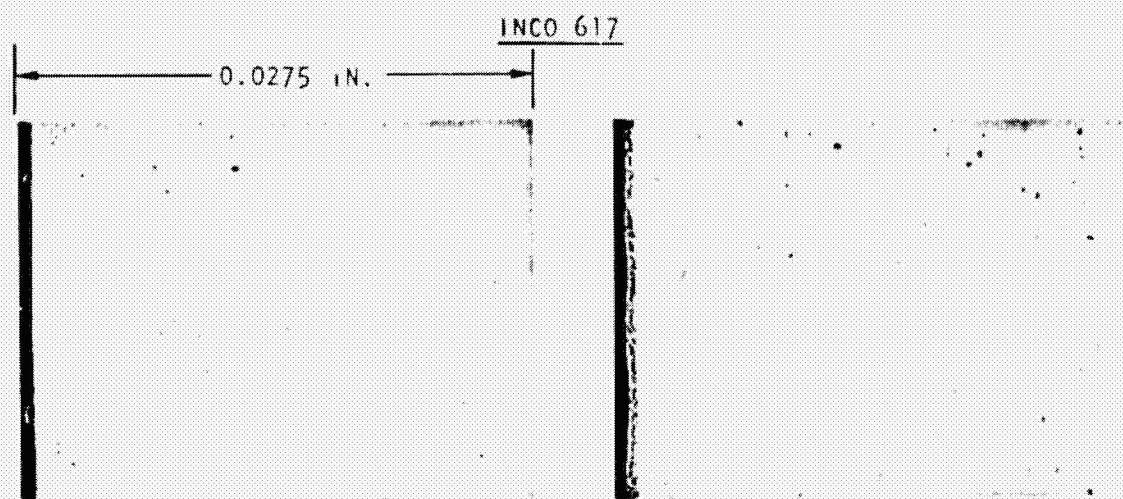


100-HOURS EXPOSURE



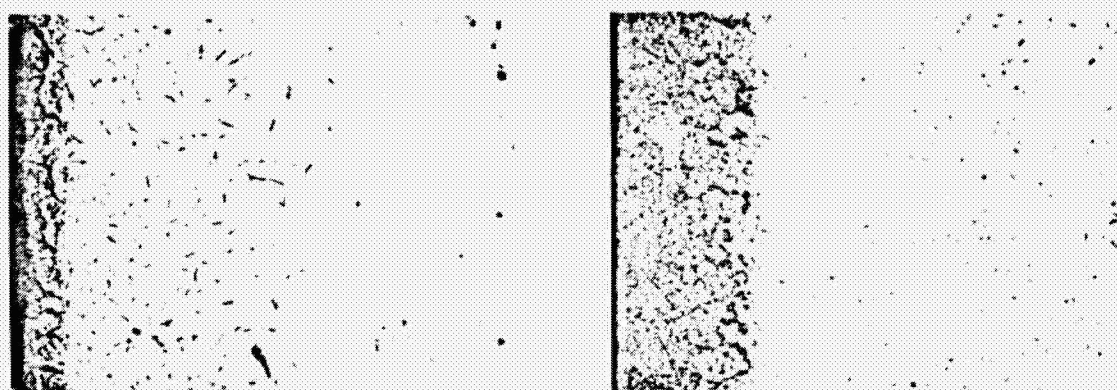
100-HOURS EXPOSURE

Figure 19. Micrograph of Test Specimen - INCO 600



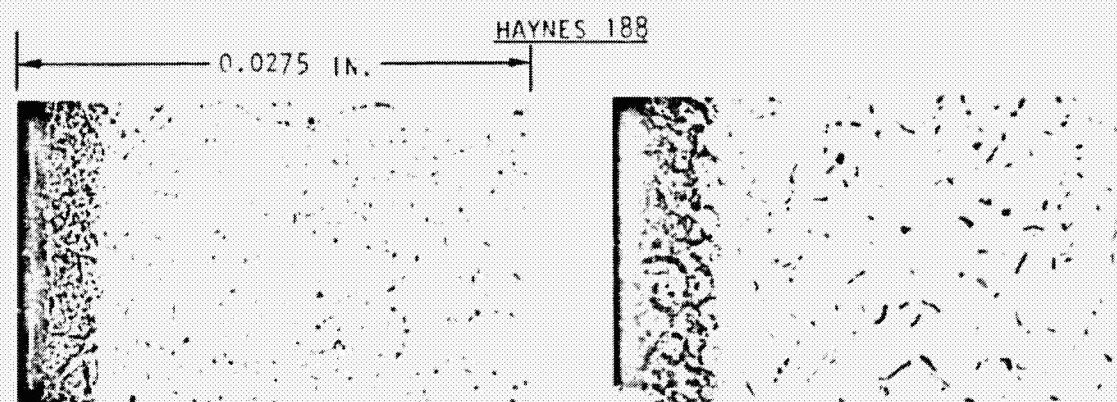
0-HOUR EXPOSURE

10-HOUR EXPOSURE



100-HOUR EXPOSURE

1000-HOUR EXPOSURE



100-HOUR EXPOSURE

1000-HOUR EXPOSURE

Figure 20. Micrograph of Test Specimen Tensile Specimens Exposed to 1800 F Nitriding Environment, Magnification - 100X

exposure. After 10 hours the affected depth was only 0.001 inch. Figure 20 compares INCO 617 with Haynes 188. The nitride appears to have penetrated the Haynes 188 grain structure more severely than the INCO 617 after both 100-hours and 1000-hours exposure.

Low-cycle fatigue information was computed from the reduction of area and ultimate strength data obtained during the tensile tests. A measure of the low-cycle fatigue characteristic is obtained by relating strain range per stress cycle ($\Delta\epsilon$) to the number of cycles to failure (N_f). An increase in the strain range reduces the number of cycles to failure in accordance with the following relations:

$$\Delta\epsilon = \Delta\epsilon_p + \Delta\epsilon_e \quad (1)$$

where $\Delta\epsilon$ = total strain range
 $\Delta\epsilon_p$ = plastic strain range
 $\Delta\epsilon_e$ = elastic strain range

$$\Delta\epsilon_p = \left(\frac{D}{N_f} \right)^{0.6} \quad \text{Manson Coffin Law (2)}$$

$$\Delta\epsilon_e = \frac{3.5\sigma_U}{E(N_f)^{0.12}} \quad \text{Basquin Law (3)}$$

where D = $100/(100-RA)$, fracture ductility
 RA^* = reduction area, percent
 N_f = cycles to failure
 σ_U^* = ultimate strength
 E = Young's module.

* RA and σ_U obtained from tensile test data.

The 0-, 10-, and 100-hour tensile test data for the Haynes 188, INCO 600, and L605 specimens were used to generate low-cycle fatigue curves in Fig.21 through 23. The INCO 600, with zero hours of nitriding exposure, is capable of 750 cycles of operation before failure, each cycle incorporating a 3-percent strain range. After 100 hours of exposure to an 1800 F nitriding environment, failure will result after only 250 such cycles. In comparison, the L605, after 100-hours exposure, is capable of 155 cycles of 3-percent strain range before failure; and the Haynes 188, after 100 hours, is capable of 85 cycles of 3-percent strain range before failure.

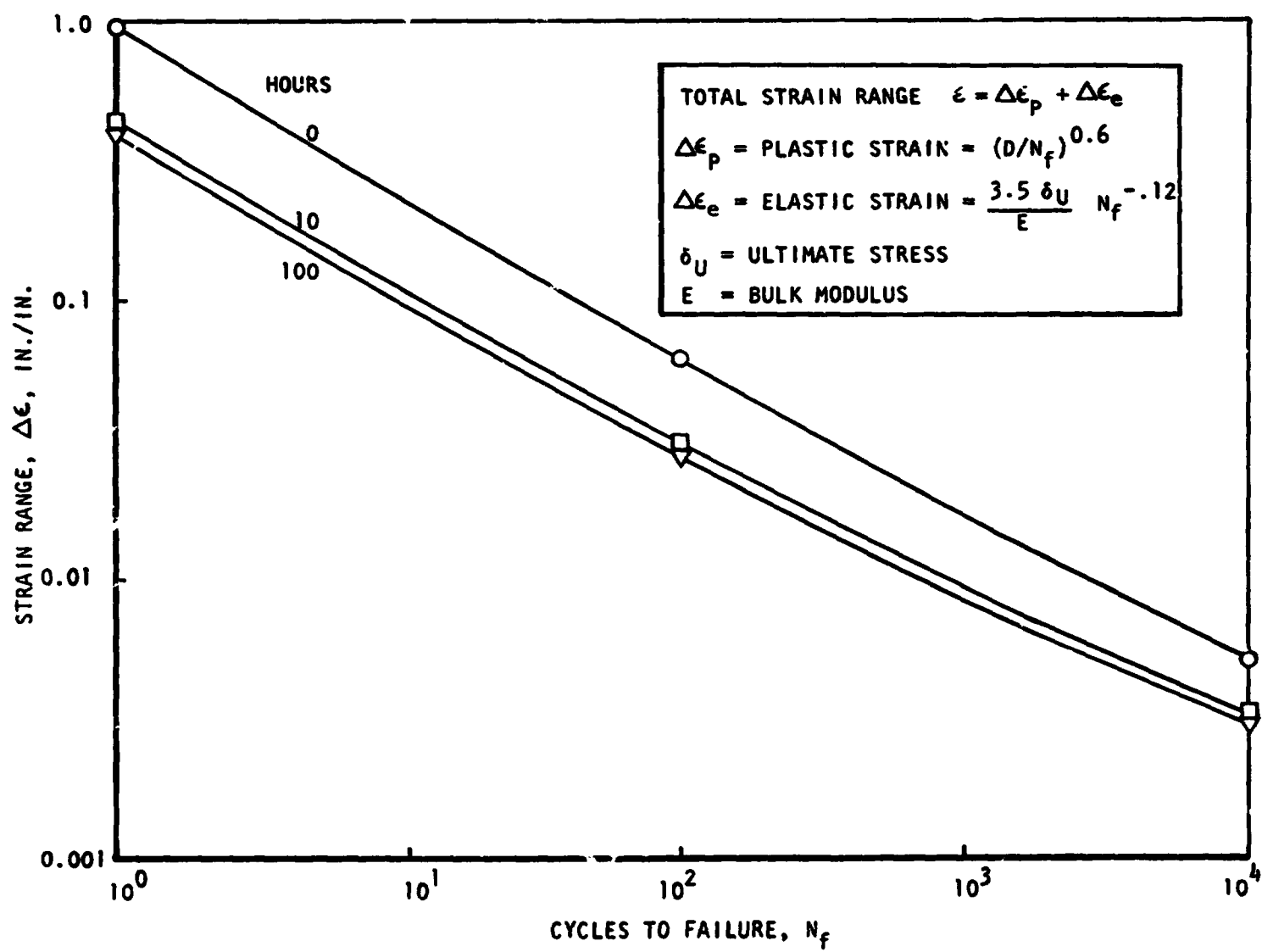


Figure 21. Material Nitriding Experiment - Haynes 188, 1800 F

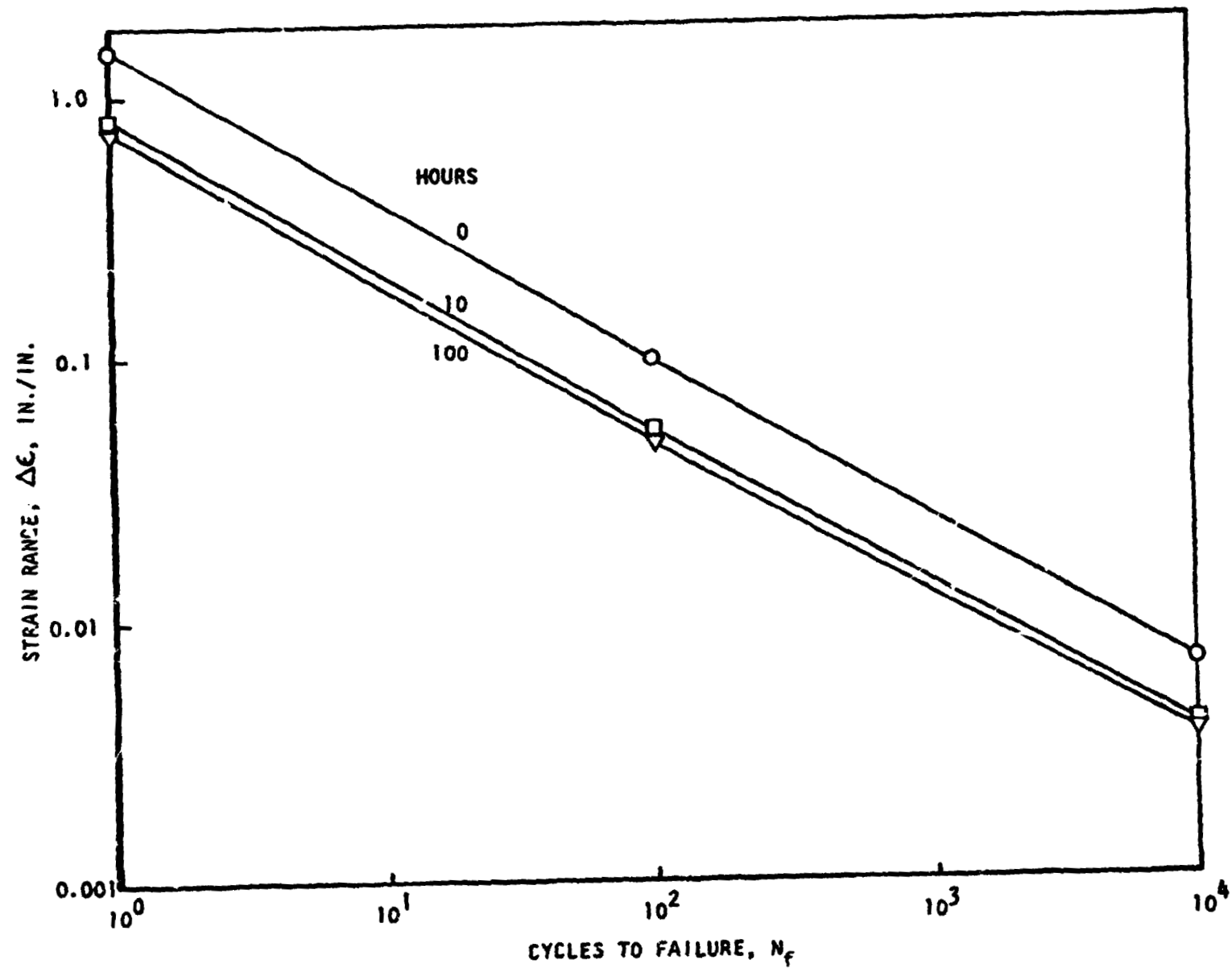


Figure 22. Material Nitriding Experiment - INCO 600, 1800 F

R-9196
39/40

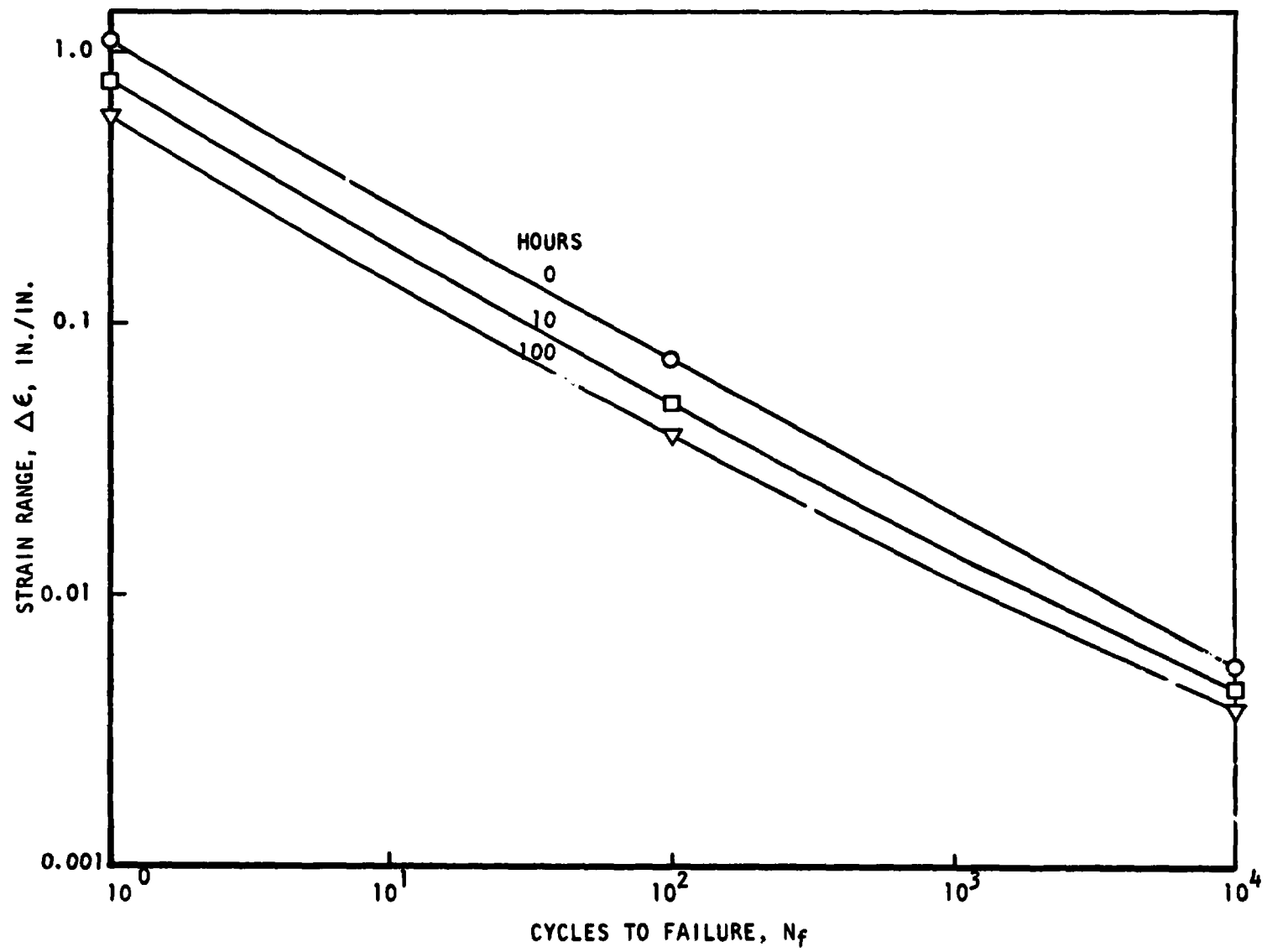


Figure 23. Material Nitriding Experiment - L605, 1800 F

APPENDIX A

TENSILE SPECIMEN WEIGHT AND THICKNESS MEASUREMENT

10 HR AND 100 HR
ON TENSILE

NITRIDATION TESTS EFFECTS
SAMPLES

10 HR TEST	WEIGHT CHANGE				THICKNESS CHANGE (MIDDLE)			
	PRETEST gms	POST TEST gms	CHANGE gms	CHANGE %	PRETEST inch	POST TEST inch	CHANGE inch	CHANGE %
L 605	19.4993	19.5510	0.0517	0.265	0.0603	0.0608	0.0005	0.83
HAYNESS 188	16.4897	16.5328	0.0437	0.265	0.0524	0.0528	0.0004	0.76
INCONEL 617	15.1052	15.1452	0.0400	0.265	0.0509	0.0513	0.0004	0.79
INCONEL 600	14.6227	14.6474	0.0247	0.169	0.0492	0.0496	0.0004	0.81
NICKEL 270	15.9920	15.9906	-0.0014	-0.00875	0.0544	0.0564	0.0020 ^(*)	3.68 ^(*)
100 HR TEST								
L 605	19.0942	19.2622	0.1680	0.88	0.0602	0.0615	0.0013	2.16
HAYNES 188	16.2584	16.4185	0.1601	0.99	0.0520	0.0533	0.0013	2.50
INCONEL 617	15.2132	15.3483	0.1351	0.89	0.0513	0.0524	0.0011	2.14
INCONEL 600	14.6129	14.7016	0.0887	0.61	0.0492	0.0499	0.0007	1.42
NICKEL 270	16.8912	(*)	(*)	(*)	0.0556	(*)	(*)	(*)

(*) POST 100HR Ni 270 SAMPLE COULD NOT BE ACCURATELY MEASURED OR WEIGHED DUE TO IRREMOVABLE NATURE OF SUPPORT BOLTS

(*) Ni 270 POST 10HR TEST THICKNESS MEAS. IS SUSPECT DUE TO SLIGHT BEND

Donald M. Gates 10/18/72
DONALD M. GATES
CHEMISTRY LABORATORY TECHNICIAN

James E. Mars
JAMES E. MARS
MANAGER CHEMICAL LABORATORIES

100 HR NITRIDATION TEST EFFECTS ON
SECONDARY CANDIDATE TENSILE SAMPLES

	WEIGHT CHANGE				THICKNESS CHANGE			
	PRETEST gms	POST TEST gms	CHANGE gms	CHANGE %	PRETEST INCH	POST TEST INCH	CHANGE INCH	CHANGE %
MULTIMET-B ^(*)	15.2758	15.5542	0.2784	1.82	.0524	.0543	.0019	3.63
TD NICKEL-B	16.1369	16.1600	0.0231	0.14	.0517	.0522	.0005	0.97
M 509 -B	16.3248	16.4482	0.1234	0.76	.0529	.0530	.0006	1.15
M 246 -B	15.7294	15.7642	0.0348	0.22	.0524	.0527	.0003	0.57

(*) THE MULTIMET-B SAMPLE WAS BROKEN DURING DISASSEMBLY OF THE BOLTED-TOGETHER SAMPLE STACK, WHEN A NICK SWIV, BEING USED TO REMOVE A BOLT, TAPPED AGAINST IT. THE BREAK OCCURRED AS A SUDDEN SNAP AND WAS NOT PRECEDED BY NOTICABLE BENDING OF THE SAMPLE.

Donald M. Gates
DONALD M. GATES
CHEMISTRY LABORATORY TECHNICIAN

James E. Mairs
JAMES E. MAIRS
MANAGER, CHEMISTRY LABORATORIES

POST 1000HR NITRIDATION TEST -- EFFECTS DATA

NOV. 30, 1972

MATL	SAMPLE TYPE	ORIGINAL WT (gms)	Final WT (gms)	CHANGE (gms)	CHANGE (%)	ORIGINAL TENSILE (mils)	Final Tensile (mils)	CHANGE (mils)	CHANGE %	EXPOSURE TIME - HR
INCO 600	Tensile #4	14.6971	14.9032 ^(a)	+0.2061 ^(a)	+1.40 ^(a)	0.0492	0.0558	+0.0066 ^(a)	+13.4 ^(a)	1000
NICKEL 270	Tensile #4	16.4499	16.6310	+0.1811	+1.10	0.0599	0.0639	+0.0040	+17.46 ^(a)	↑
HAYNES 188	Tensile #4	16.4325	16.8402	+0.4077	+2.48	0.0520	0.0568	+0.0048	+9.23	↓
INCO 617	Tensile #4	15.5270	15.8737	+0.3467	+2.23	0.0515	0.0542	+0.0027	+5.24	1000
MULTIMET-A	Tensile #4	15.1204	15.8784	+0.7580	3.43	0.0520	0.0568	+0.0048	+9.23	397.25
TD NICKEL-A	Tensile #4	15.9672	16.4830 ^(a)	(a)	(a)	0.0508	0.0527	+0.0019	+3.74	↑
INCO 617-Chrome Plated - Uniaxial A	Tensile #4	16.3380	16.2334 ^(a)	(a2)(a5)	(a2)(a5)	0.0585	0.0580	(a2)	(a2)	↑
INCO 617-Chrome Plated - Biaxial A	Tensile #4	16.6099	16.3315 ^(a)	(a2)(a5)	(a2)(a5)	0.0607	0.0588	(a2)	(a2)	↑
M-246-A	Tensile #4	15.8950	15.9122	+0.0172	+0.42	0.0525	0.0534	+0.0009	+1.71	↓
M-509-A	Tensile #4	16.1549	16.4107	+0.2558	+1.52	0.0522	0.0556	+0.0034	+6.51	397.25
INCO 600	Screen - 764 NICH	7.6326	7.9020	+0.2694	+3.53	↑				1000
	Screen - 1 NICH	7.6467	7.9059	+0.2592	+3.39	DATA NOT TAKEN				↑
	Screen - 2 NICH	7.5814	7.8531	+0.2717	+3.58					
	Screen - 3 NICH	7.5249	7.7613	+0.2364	+3.14					↓
INCO 600	Screen Pack	95.5301	92.4075 ^(a)	+1.8614 ^(a)	+1.96	0.4538	0.4771	+0.0233	+5.13	1000

James E. Hays
MANAGER, CHEMISTRY

Donald M. Gifford
CHEMISTRY WORKS
TECHNICIAN

ORIGINAL PAGE IS
OF POOR QUALITY

POST 1000HR NITRIDATION TEST EFFECTS DATA

NOV 30, 1972

NOTES (#1) THE BOLTS AND NUTS RETAINING THE TD-NICKEL TENSILE SAMPLE LEFT A residual cake of heavily nitrided material in the mounting holes at each end of the sample. The weight given includes the contribution of this material.

(#2) CHROME PLATING ON SAMPLES CAME OFF DURING 1000HR EXPOSURE PERIOD OF 24000V CANDIDATE SAMPLES. OBSERVATION OF ABSENCE OF CHROME PLATING WAS MADE WHEN FURNACE WAS OPENED AT COMPLETION OF 1000HR TEST

(#4) INFO 600 SAMPLE EXPOSED FOR 1000HRS HAD SURFACE FLAKES (DARK IN COLOR) AT END OF TEST AND SOME OF THESE FLAKES WERE ON THE OUTEN OF THE QUALITY TUBE. A FINE FILAMENT OR CILIA TYPE GROWTH AT THE UNSTREAM EDGE OF THE INFO 600 AND NICKEL 270 SAMPLES WAS OBSERVED. THIS GROWTH WAS NOT OBSERVED ON OTHER SAMPLES.

(#5) TOTAL WEIGHT OF ALL FLAKES- 0.829g

(#7) TOTAL SCREEN PACK WT. LOSS FROM VIBRATION: 0.0088gms.

(#9) LARGE MEASURED THICKNESS CHANGE PROBABLY DUE TO NONUNIFORM SURFACE.

(#10) LARGE MEASURED THICKNESS CHANGE PROBABLY DUE TO DISTORTED SAMPLE SHAPE.

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OF POOR QUALITY

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NOV 4

APPENDIX B

TENSILE TEST DATA

AS REC

ROCKETDYNE
A DIVISION OF NORTH AMERICAN ROCKWELL CORPORATION
MATERIALS AND PROCESSES DEPT

TENSILE TEST DATA REPORT

MATERIAL GAS GEN CONDITION _____ LABORATORY NO 7210-71
SPECIFICATION _____ DATE SUBMITTED 10-19-72
JOB DESCRIPTION 1800°F ARGON
CHARGE NO 03711-09444-04300 SUBMITTED BY MARCY

Primary Candidates - Whetstone

MAXIMUM REQUIREMENTS								ELONG		ELONG	
MINIMUM REQUIREMENTS											
BAR NO	SIZE IN	AREA SQ IN	YIELD LOAD LBS	YIELD STR (0.2% OFFSET) KSI	ULTIMATE LOAD LBS	ULTIMATE STRENGTH KSI	ELONG. ATION % 1/2	REDUCTN OF AREA 1/2	HARD. NESS 1/2	NOTE	
				<u>1800 F</u>							
<u>HAYNES</u>											
<u>188</u>	<u>252X052</u>	<u>0131</u>	<u>302</u>	<u>23.1</u>	<u>362</u>	<u>27.6</u>	<u>92.0</u>	<u>38.0</u>	<u>92.0</u>	<u>60.0</u>	
<u>INCO</u>											
<u>617</u>	<u>251X051</u>	<u>0128</u>	<u>89</u>	<u>7.0</u>	<u>148</u>	<u>11.6</u>	<u>80.0</u>	<u>53.0</u>		<u>35.0</u>	
<u>605</u>	<u>249X050</u>	<u>0148</u>	<u>259</u>	<u>17.5</u>	<u>319</u>	<u>21.6</u>	<u>120.0</u>	<u>84.0</u>	<u>96.0</u>	<u>55.0</u>	
<u>INCO</u>											
<u>600</u>	<u>250X049</u>	<u>0128</u>	<u>86</u>	<u>7.0</u>	<u>125</u>	<u>10.2</u>	<u>148.0</u>	<u>98.0</u>	<u>75.0</u>	<u>60.0</u>	
<u>NI</u>											
<u>270</u>	<u>252X053</u>	<u>0134</u>	<u>29</u>	<u>2.2</u>	<u>54</u>	<u>4.0</u>	<u>128.0</u>	<u>82.0</u>	<u>30.0</u>	<u>51.0</u>	
		<u>RA</u>									
<u>188</u>	<u>57.8</u>										
<u>617</u>	<u>35.2</u>										
<u>605</u>	<u>64.5</u>										
<u>600</u>	<u>87.0</u>										
<u>270</u>	<u>85.7</u>										

COMMENTS:

ODE: (F) DENOTES FLAW IN BAR
(O) BAR BROKE OUTSIDE GAGE MARK
(G) BAR BROKE AT GAGE MARK
(E) DENOTES ERRATIC CURVE

REPORTED BY Pacific DATE 10-20-72
HOURS EXPENDED 15 HRS
APPROVED BY _____ DATE _____

ROCKETDYNE
A DIVISION OF NORTH AMERICAN ROCKWELL CORPORATION
MATERIALS AND PROCESSES DEPT.

TENSILE TEST DATA REPORT

MATERIAL GAS GEN. CONDITION _____ LABORATORY NO 7210-71
SPECIFICATION _____ DATE SUBMITTED 10-14-72
JOB DESCRIPTION 1800 F ALCON
CHARGE NO 03711-09444-04300 SUBMITTED BY MARCY

Primary Candidates - 12 Hq

MAXIMUM REQUIREMENTS											
MINIMUM REQUIREMENTS								Elong		Elong	
BAR NO.	SIZE IN	AREA SQ IN	YIELD LOAD LBS	YIELD STR (0.2% OFFSET) KSI	ULTIMATE LOAD LBS	ULTIMATE STRENGTH KSI	ELONGATION % GL	REDUCTN OF AREA %	HARDNESS RB	NOTE	
				1800F							
HAYNES											
188	252x0525	0.132	290	22.0	368	27.9	56.0	36.0	93.0	25.0	
INCO											
600	251x0445	0.124	91	7.3	134	10.8	68.0	48.0	60.0	37.0	
L											
605	252x0605	0.152	334	22.0	395	26.0	56.0	54.0	99.0	37.0	
INCO											
617	253x051	0.129	186	14.4	243	18.8	56.0	40.0	87.0	31.0	
NI											
270	254x055	0.140	24	1.7	27	1.9	16.0	10.0	*	6.0	
		RA									
188		22.4									
600		44.1									
605		48.0									
617		32.6									
270		9.0									

COMMENTS: * HARDNESS 15T 54.0

CODE: (F) DENOTES FLAW IN BAR
(O) BAR BROKE OUTSIDE GAGE MARK
(G) BAR BROKE AT GAGE MARK
(E) DENOTES ERRATIC CURVE

REVIEWED BY Karl Lee DATE 10-23-
HOURS EXPENDED 10 HRS
APPROVED BY _____ DATE _____

100 HRS

ROCKETDYNE
A DIVISION OF NORTH AMERICAN ROCKWELL CORPORATION
MATERIALS AND PROCESSES DEPT

TENSILE TEST DATA REPORT

MATERIAL GAS GEN CONDITION _____ LABORATORY NO 7210-71
SPECIFICATION _____ DATE SUBMITTED 10-19-72
JOB DESCRIPTION 1800 FABRON
CHARGE NO 03711-09444-04300 SUBMITTED BY MARCY

Primary Candidates - 100 H2

MAXIMUM REQUIREMENTS								ELONG		ELONG	
MINIMUM REQUIREMENTS								ELONG		ELONG	
BAR NO.	SIZE IN.	AREA SQ IN	YIELD LOAD LBS	YIELD STR 10 2% OFFSET KSI	ULTIMATE LOAD LBS	ULTIMATE STRENGTH KSI	ELONGATION % GL	REDUCTN OF AREA %	HARDNESS RB	NOTE	
				1800 F							
HAYNES											
188	254X053	0.135	308	22.8	377	27.9	38.0	25.0	97.0	18.0	
INCO											
600	249X050	0.125	110	8.8	155	12.4	52.0	56.0	62.0	44.0	
L											
105	246X061	0.151	417	27.6	427	28.3	56.0	40.0	*	32.0	
2 NCO											
617	251X052	0.131	252	19.2	297	22.7	36.0	23.0	91.0	21.0	
N1											
270	253X057	0.144	21	1.5	45	3.1	76.0	50.0	**	38.0	
		RA									
188		18.5									
600		51.0									
605		31.8									
617		20.6									
270		79.9									

COMMENTS: * HARDNESS ON L 605 PC-22
** HARDNESS ON N1270 T42

CODE: (F) DENOTES FLAW IN BAR
(O) BAR BROKE OUTSIDE GAGE MARK
(G) BAR BROKE AT GAGE MARK
(E) DENOTES ERRATIC CURVE

REPORTED BY Pacchias - 1024
HOURS EXPENDED 1.5
APPROVED BY P.T. Foster DATE 12-13-72

ROCKETDYNE
A DIVISION OF NORTH AMERICAN ROCKWELL CORPORATION
MATERIALS AND PROCESSES DEPT

TENSILE TEST DATA REPORT

MATERIAL GAS GENERATOR CONDITION _____ LABORATORY NO 7211-104
SPECIFICATION _____ DATE SUBMITTED 11-29-72
JOB DESCRIPTION TENSILE, 1900°F
CHARGE NO 03711-09444-04300 SUBMITTED BY MARLY

22A candidates - unexposed & 100 HRS

MAXIMUM REQUIREMENTS										
MINIMUM REQUIREMENTS										
BAR NO.	SIZE IN	AREA SQ IN	YIELD LOAD LBS	YIELD STR (0.2% OFFSET) KSI	ULTIMATE LOAD LBS	ULTIMATE STRENGTH KSI	ELONGATION % GL	REDUCTN OF AREA %	HARDNESS	NOTE
<u>UNEXPOSED</u>										
M509	.052X.249	.0129	340	26.3	415	32.2	26.0	22.4		RC = 26.0
TDN1	.051X.249	.0127	43	3.4	57	4.5	34.0	21.2		RB = 96.5
Multimet	.051X.249	.0127	213	16.8	247	23.4	29.0	46.4		RB = 89.0
M246	.052X.248	.0129	635	49.2	965	74.8	4.0	7.7		RC = 37.0 *
<u>EXPOSED</u>										
<u>100 HRS</u>										
M509 B	.053X.251	.0133	370	27.8	495	37.2	14.0	10.5		RC = 33.0
TDN1 B	.052X.250	.0131	32	2.4	45	3.4	41.0	30.5		RB = 96.0 (G)
M246 B	.052X.246	.0129	755	58.5	995	77.1	6.0	4.6		RC = 35.5

COMMENTS: * EXTRAPOLATED CURVE

CODE: (F) DENOTES FLAW IN BAR
(O) BAR BROKE OUTSIDE GAGE MARK
(G) BAR BROKE AT GAGE MARK
(E) DENOTES ERRATIC CURVE

REPORTED BY P. Kildan DATE 12-8-72
HOURS EXPENDED 20
APPROVED BY _____ DATE _____

ROCKETDYNE
A DIVISION OF NORTH AMERICAN ROCKWELL CORPORATION
MATERIALS AND PROCESSES DEPT

TENSILE TEST ATA REPORT

MATERIAL GAS GENERATOR CONDITION _____ LABORATORY NO 2211-104
SPECIFICATION _____ DATE SUBMITTED 11-24-72
JOB DESCRIPTION Tensile 1800°F 1000 HR.
CHARGE NO Q3711-09444-09300 SUBMITTED BY MZRCY

PRIMARY CANDIDATES - 1000 HRS

MAXIMUM REQUIREMENTS										
MINIMUM REQUIREMENTS										
BAR NO	SIZE IN	AREA SQ IN	YIELD LOAD LBS	YIELD STR 10 2% OFFSET KSI	ULTIMATE LOAD LBS	ULTIMATE STRENGTH KSI	ELONGATION % 1 GL	REDUCTN OF AREA %	HARDNESS	NOTE
<u>1000 HRS</u>										
<u>HAYNESIES</u>	<u>250X.056</u>	<u>.0144</u>	<u>333</u>	<u>23.1</u>	<u>493</u>	<u>34.2</u>	<u>7.5</u>	<u>6.3</u>	<u>RB</u>	<u>= 99.0</u>
<u>#2 Post</u>										
<u>INCO 600</u>										
<u>#2 Post</u>	<u>254X.054</u>	<u>.0137</u>	<u>111</u>	<u>8.1</u>	<u>166</u>	<u>12.1</u>	<u>14.0</u>	<u>40.6</u>	<u>RB</u>	<u>= 54.5</u>
<u>INCO 617</u>										
<u>#2 Post</u>	<u>254X.053</u>	<u>.0135</u>	<u>290</u>	<u>21.5</u>	<u>415</u>	<u>30.7</u>	<u>18.0</u>	<u>16.3</u>	<u>RB</u>	<u>= 93.5</u>
SECONDARY CANDIDATES - 400 HRS										
<u>509 #6</u>										
<u>Post</u>	<u>251X.055</u>	<u>.0138</u>	<u>368</u>	<u>26.7</u>	<u>470</u>	<u>34.1</u>	<u>12.0</u>	<u>8.0</u>	<u>RC</u>	<u>= 35.5</u>
<u>Exposure Time</u>										
<u>TD Ni</u>										
<u>#6 Post</u>	<u>252X.053</u>	<u>.0134</u>	<u>415</u>	<u>3.6</u>	<u>49</u>	<u>3.6</u>	<u>38.0</u>	<u>58.9</u>	<u>RB</u>	<u>= 74.0</u>
<u>Exposure Time</u>										
<u>Multimet</u>					<u>324</u>					
<u>#6 Post</u>	<u>257X.056</u>	<u>.0144</u>	<u>226</u>	<u>15.7</u>	<u>374</u>	<u>26.0</u>	<u>52.0</u>	<u>59.7</u>	<u>RB</u>	<u>= 91.5</u>
<u>Exposure Time</u>										
<u>M246 #6</u>	<u>249X.053</u>	<u>.0132</u>	<u>400</u>	<u>45.4</u>	<u>523</u>	<u>69.0</u>	<u>2.0</u>	<u>2.2</u>	<u>RC</u>	<u>= 31.0</u>
<u>Post Exposure</u>	<u>*</u>									

COMMENTS: * Extrapolated Curve

CODE: (F) DENOTES FLAW IN BAR
(O) BAR BROKE OUTSIDE GAGE MARK
(G) BAR BROKE AT GAGE MARK
(E) DENOTES ERRATIC CURVE

REPORTED BY S. W. Liden DATE 12-17-72
HOURS EXPENDED 17
APPROVED BY R. T. Heaster DATE 12-18-72

APPENDIX C

HARDNESS PENETRATIVE TESTS

DATA SHEET

7th No. 2-1037

Inco 600

500 gram load 10.25 obj 3.400

J.R. Gierstmaier

From edge apex	Film units	Re Equiv	RB Equiv		From edge apex	Film units	Re Equiv	RB Equiv	
Inco 600	100	hrs exposure			Inco 600	0	hrs exposure		
0019	370		99.6	0.491	0019	470		76.0	15.1
0039	386		94.7	Thick	0039	458		78.8	Thick
0058	396		92.6		0058	452		80.2	
0077	428		85.8		0077	480		73.3	
0096	450		80.6		0096	458		78.8	
0115	446		81.5		0115	464		77.5	
0137	468		76.5		0137	456		79.3	
0157	450		80.6		0157	456		79.3	
0177	465		77.3		0177	474		74.7	
0197	448		81.0						
0236	446		81.5		0256	474		74.7	16.4/10.7
0256	468		76.5	half way	0295	468		76.5	00.39 52.1

			RB	
Inco 600	10	hrs exposure		
0019	430		85.4	0.471
0039	425		86.5	Thick
0058	440		83.0	
0077	468		76.5	
0096	460		78.4	
0115	454		79.7	
0137	464		77.5	
0157	470		76.0	
0177	478		73.7	
0197	472		75.3	
0236	462		78.0	half way
0275	448		81.0	as per

DATA SHEET

mt no. 2-1038

Jr. Gerstner

500 gram Load 10.25 Obj. Ticks

Dist from Filan
to L 605 (100 hrs)
Re Equin
RB Equin

0019	328	30.0	} Thick
0039	318	32.7	
0058	322	31.7	
0077	326	30.7	
0096	337	27.7	
0115	342	26.5	
0137	334	28.5	
0157	332	29.0	
0177	326	30.7	
0197	326	30.7	
0216	338	27.5	} Half way
0236	328	30.0	
0256	332	29.0	
0275	324	31.3	
0295	326	30.7	

L 605 (10 hrs) 0.587

0019	290	39.7	} Thick
0039	340	27.0	
0058	318	32.7	
0077	338	27.5	
0096	334	28.5	
0115	336	28.0	
0137	352	23.7	
0157	358	22.0	
0177	356	22.5	
0197	352	23.7	
0216	350	24.2	} Half way
0236	348	24.7	
0256	350	24.2	
0295	348	24.7	

Dist from Filan
to L 605 (0 hrs)
Re Equin
RB Equin

0019	384	95.2	} Thick
0039	392	93.4	
0058	384	95.2	
0077	382	95.7	
0096	386	94.7	
0115	384	95.2	
0137	396	92.6	
0157	386	94.7	
0177	400	91.8	
0216	426	86.3	
0256	400	91.8	} Half way

DATA SHEET

mt. 2-1039

500 gram Load 10.25 06.10 10.25

John H. Long

Time	RC	RB	Time	RC	RB
Hydro 158 Long	Hydro 158 Long	Hydro 158 Long	Hydro 158 Long	Hydro 158 Long	Hydro 158 Long
002 288 40.2	001 36.5 20.2		002 340 27.0		
004 278 42.6	004 36.5 20.2		006 350 24.2		
006 303 36.5	008 358 22.0		010 367 98.3		
008 340 27.0	012 372 95.2		014 384 91.8		
010 333 28.7	016 367 96.7		040 372 98.3		
012 330 29.7					
014 364 20.5					
016 345 25.7					
018 356 22.5					
020 364 20.5					
022 350 24.2					
024 340 27.0					
040 348 24.7					
001 300 37.2					
002 342 26.5					
004 340 27.0					
006 348 24.7					
008 354 23.0					
010 352 23.7					
012 360 21.5					
014 360 21.5					
016 362 21.0					
024 377 97.0					
040 374 97.7					